Corrosion

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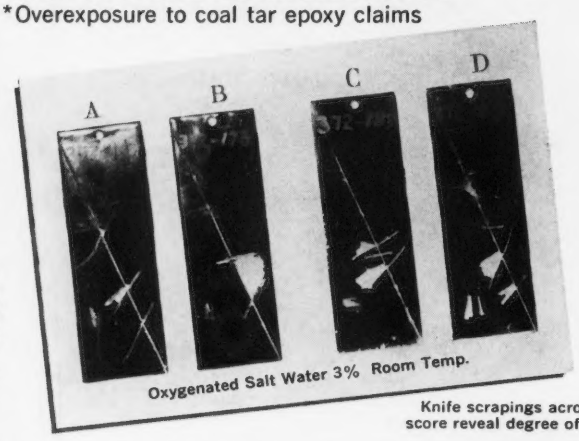
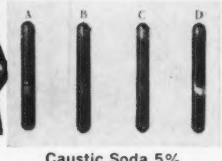
PROTECTIVE INDUSTRIES
NATIONAL ASSOCIATION OF CORROSION ENGINEERS
ORGAN SEC. 543

SUFFERING FROM COALTAR EPOXICATION?*

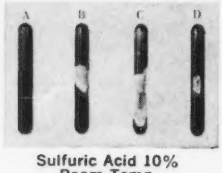
*Overexposure to coal tar epoxy claims



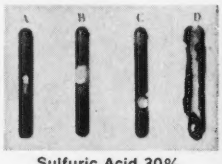
Knife scrapings across diagonal score reveal degree of undercutting.



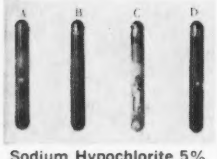
Caustic Soda 5% Room Temp.



Sulfuric Acid 10% Room Temp.

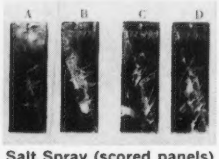


Sulfuric Acid 30% Room Temp.

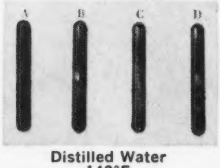


Sodium Hypochlorite 5% Room Temp.

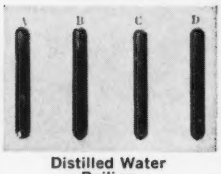
Aviation Gas 100/130 Octane Room Temp.



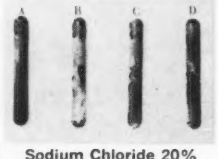
Salt Spray (scored panels) Room Temp.



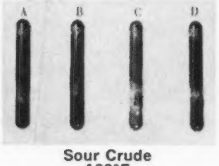
Distilled Water 140°F



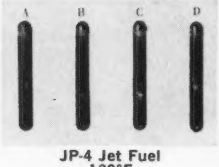
Distilled Water Boiling



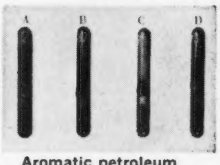
Sodium Chloride 20% Boiling



Sour Crude 120°F



JP-4 Jet Fuel 120°F



Aromatic petroleum hydrocarbon solvent—120°F

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116

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Cathodic Protection of Offshore Platforms, by W. A. Johnson and J. T. Condry

Case Histories of Differential Aeration Corrosion in Underground Telephone Cables, by D. T. Jones

Stress Corrosion Cracking of Austenitic Stainless Steel, by W. B. Brooks.

•

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Southeast Region	Aug.
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T-1M-2 Non-Ferrous Construction Materials

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T-2H-3 To Assemble and Present Service Histories of Asphalt-Type Pipe Coatings

T-2H-4 Asphalt-Type Internal Coating for Pipe Lines

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T-2H-5 To Review and Encourage Publication of Technical Articles on Asphalt-Type Protective Coatings

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T-2M-4 Corrosion of Submarine Aluminum Pipe Lines in Lake Maracaibo, Venezuela

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T-2M-5 Corosion of Submarine Aluminum Pipelines in Sea Water

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T-2M-6 Corrosion of Submarine Aluminum Pipelines in Fresh Water

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T-2M-7 Internal Corrosion Problems for Handling Oilfield Brines, Crude Oil, and Natural Gases

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T-3A-2 Methods of Testing & Screening Corrosion Inhibitors

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T-3A-3 Materials Available for and Application of Corrosion Inhibitors

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T-4C Non-Metallic Sheaths and Coatings

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T-4H Tests and Surveys of Cable Sheaths

T-4J Cathodic Protection of Cable Sheaths

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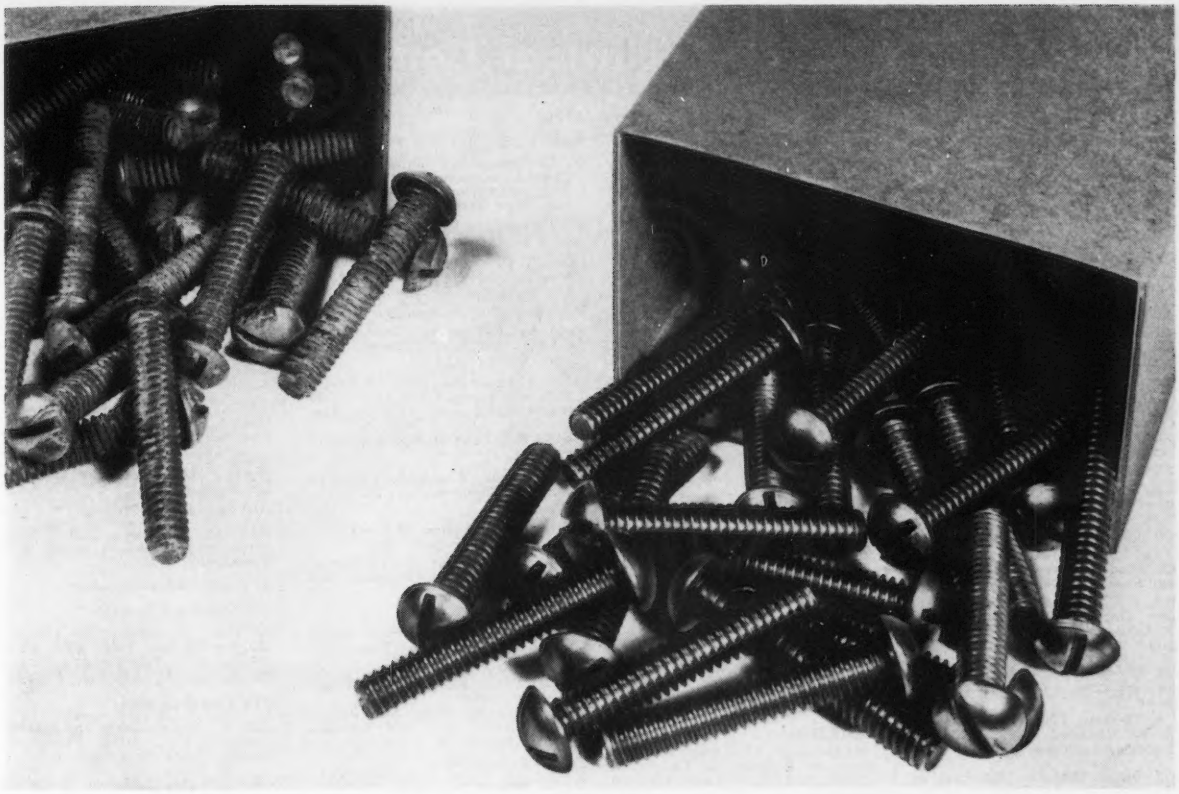

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T-6A-20 Plastisols

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T-6B-6 Modified Alkyd Varnish

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T-6B-8 Epoxy (Amine Cured)

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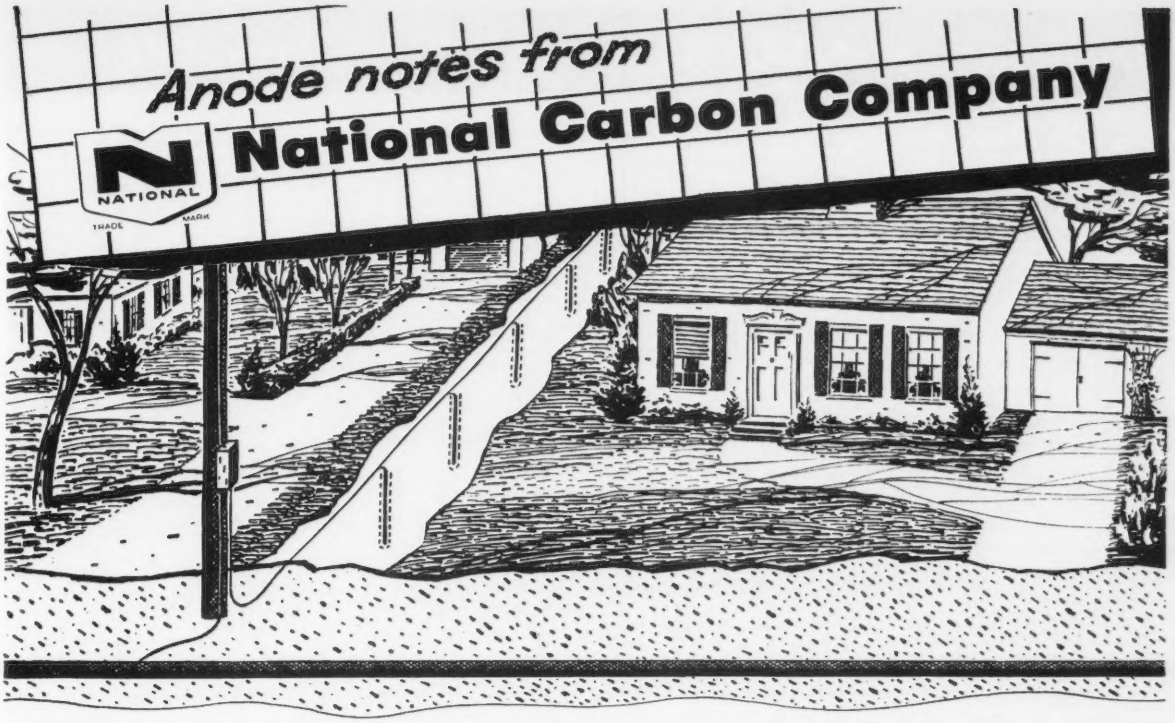


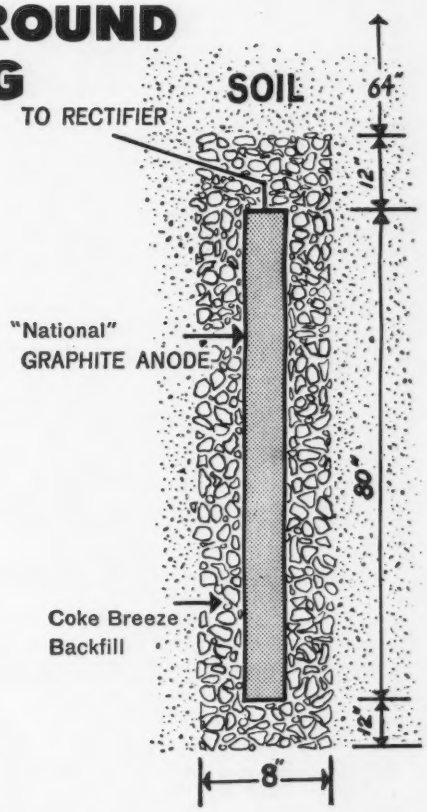
PROTECTION OF UNDERGROUND GAS DISTRIBUTION PIPING

In 1947 an anode comparison test* was sponsored by the TP-3 Subcommittee of the N.A.C.E. In this test, four beds were installed by the Mississippi Power and Light Company with various anode and backfill materials. Each year one quarter of the anodes were removed and inspected.

Based on this test, 2″ x 80″ graphite anodes in carbonaceous backfill were installed to protect the underground gas distribution lines in March, 1951. These beds have been in operation for more than 8 years with little change in current output.

In bed No. 23, 8 anodes were installed in coke breeze in 15′ deep holes spaced on 15′ centers. The original bed protected approximately 12,600′ of 2″ and 5,500′ of 3″ hot coal tar enamel field coated steel gas lines. Additional pipe has been installed in the intervening years. The bed is still putting out between 8 and 9 amps at 5-7 volts which is sufficient to give a protective potential of .9 volts to a copper — copper sulfate half cell.

*See "Final Report on Four Annual Anode Inspections" by Technical Unit Committee T-2B on Anodes for Impressed Current in CORROSION, Volume 12, 1956, Page 47t.



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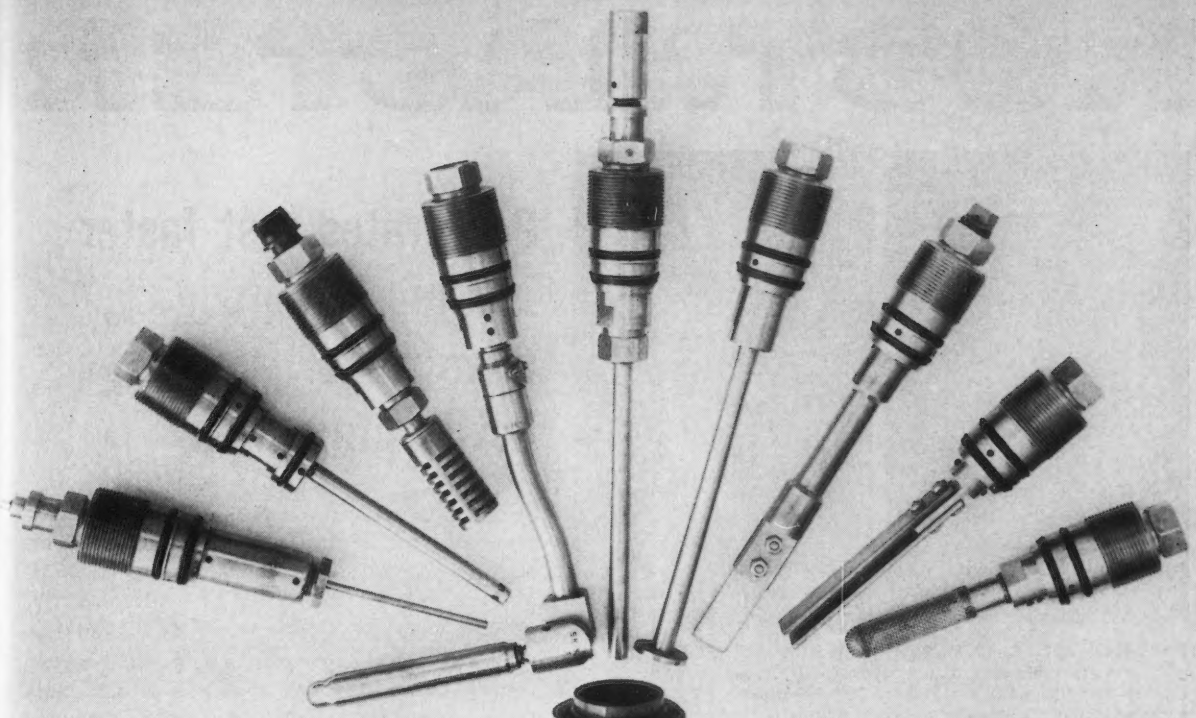

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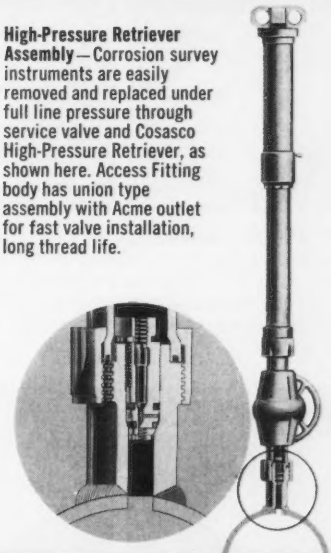

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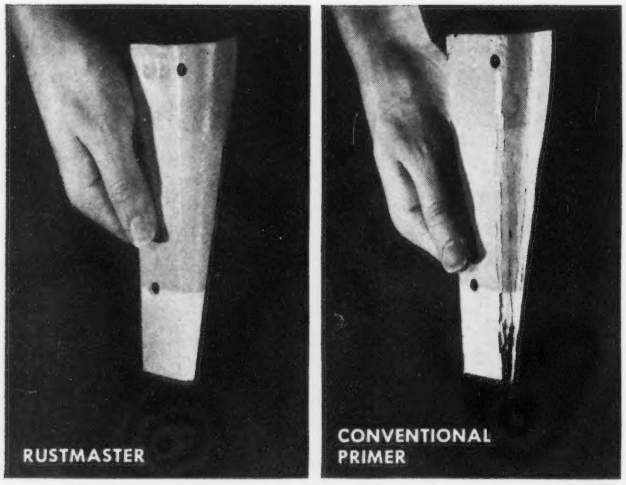

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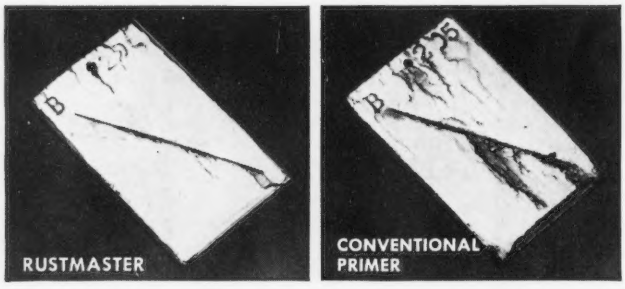

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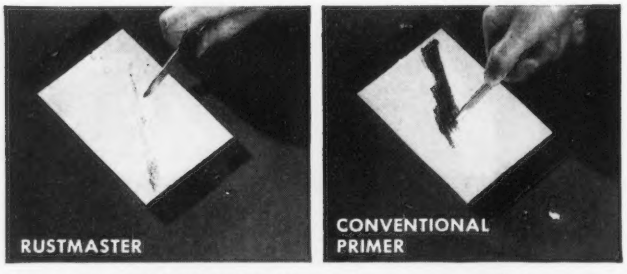

PROOF OF BETTER PROTECTION—Primers were applied to rusted metal panels and exposed to weatherometer test for 1600 hours. Scraping of both panels with knife blade showed that conventional primer pigment remained on the surface of the rust. RUSTMASTER vehicle and anti-corrosive pigments were bound into the rust, proving deeper penetration, more protection.

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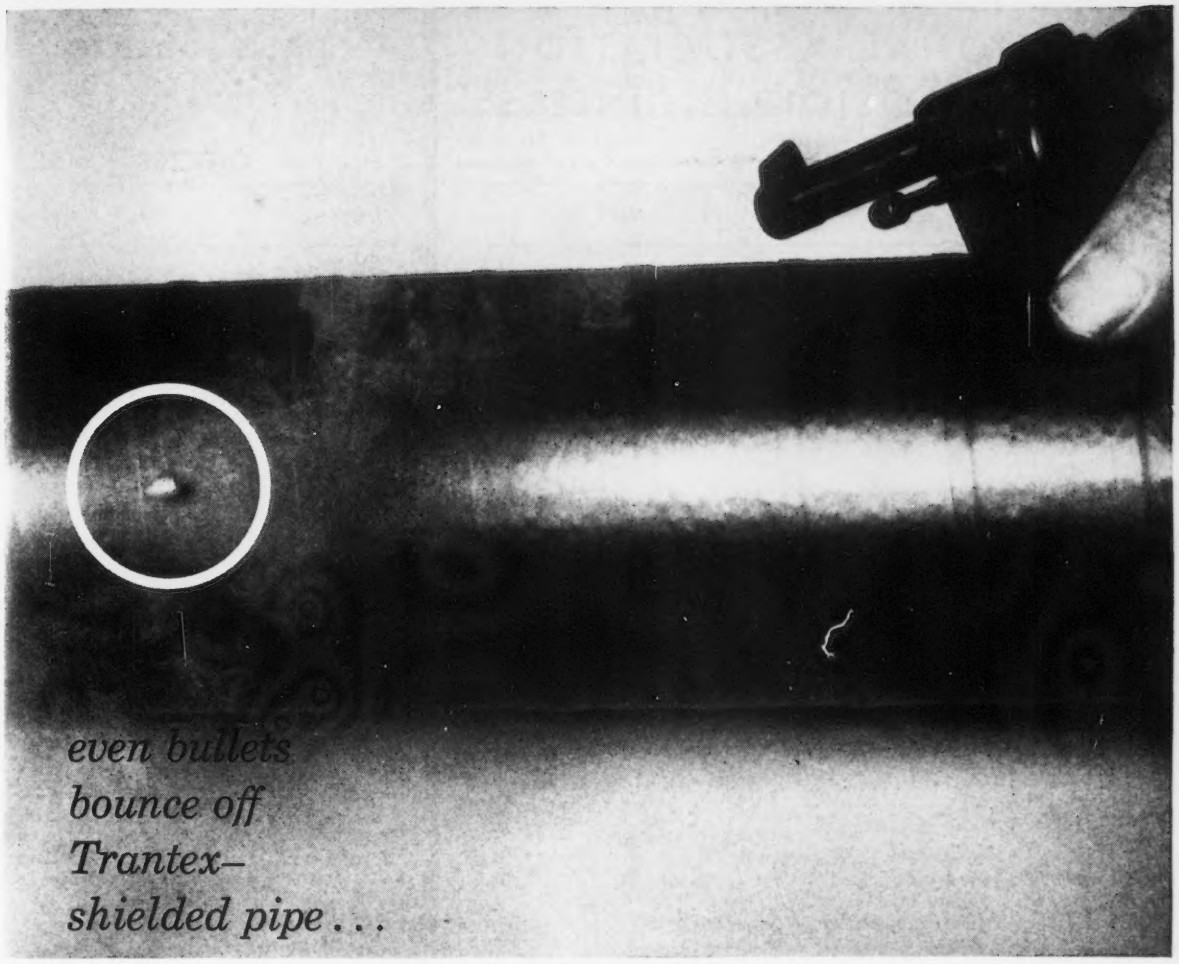

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(a) To promote the prevention of corrosion, thereby curtailing economic waste and conserving natural resources.

(b) To provide forums and media through which experiences with corrosion and its prevention may be reported, discussed and published for the common good.

(c) To encourage special study and research to determine the fundamental causes of corrosion, and to develop new or improved techniques for its prevention.

(d) To correlate study and research on corrosion problems among technical associations to reduce duplication and increase efficiency.

(e) To promote standardization of terminology, techniques, equipment and design in corrosion control.

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OFFICIAL MONTHLY PUBLICATION

National Association of Corrosion Engineers

An Association of Individuals and Organizations Concerned with Control of Corrosion

1061 M & M Building Houston 2, Texas

Vol. 15 July, 1959 No. 7

Comparisons of Wagner's Analysis of Cathodic Protection With Operating Data for Ships*

By J. H. GREENBLATT

Abstract

The predicted values of the key parameters in Wagner's analytical expression for the conditions for cathodic protection are compared with values found empirically from operating experience in the protection of ships. Agreement is good over certain ranges of the key parameters. Discrepancies in values and possible reasons for their occurrence are discussed. 5.2.4

Introduction

IN RECENT papers Wagner[1, 2] discusses the theory of cathodic protection and determines the conditions under which complete cathodic protection can be obtained for a number of models. The object of this note is to apply his criteria to systems presently fitted to Royal Canadian Navy ships to see if the general rules obtained empirically conform to Wagner's picture.

Analysis of Systems

Two basic types of cathodic protection systems are used on Royal Canadian Navy ships: controlled galvanic magnesium anode systems, and impressed current systems using consumable steel anodes or inert anodes of lead-2 percent silver alloy. Graphite and platinum anodes have been used in the second application on an experimental basis. In all cases impressed current anodes 5 inches wide for permanent anodes and 5 inches to 7 inches for steel are mounted in brackets which are themselves mounted on a painted surface or a polychloroprene current shield. The magnesium anodes, 10 inches wide are mounted on rubber sheeting 16 inches wide and insulated from the hull and grounded internally. Thus all systems used could fit Wagner's model shown in Figure 1 for an anode in the plane of the metal to be protected. In the diagram, S is the width of the insulator under the anode and C is as shown in the diagram. Figure 2 is Wagner's model for an anode at a distance from the metal to be protected. Systems whose working faces are at a distance from the metal to be protected because of bracket height or other considerations will also be analyzed according to this model to compare the general predictions of Wagner's treatment with operating experience.

By obtaining the potential distribution in the electrolyte around the cathodically protected metal, Wagner arrives at the condition for complete protection with minimum hydrogen evolution. This condition is given as follows:

$$\Delta E_{max} = \left(\frac{J_{ox}C}{\delta}\right)f \qquad (1)$$

Where f is a dimensionless function that is different for the various models considered. J_{ox} is the current density for oxygen reduction, δ the conductivity in ohms^{-1} cm^{-1}, and ΔE_{max}, the maximum variation in the local single electrode potential under conditions producing complete cathodic protection. It is defined as follows:

$$\Delta E_{max} = E_{eq} - E^*$$

Where E_{eq} is the equilibrium single electrode potential of the protected metal, E* the potential at which significant hydrogen evolution occurs.

On RCN ships, anodes are usually mounted on the bilge keels of ships and the value of $\frac{C}{2}$ ranges from 15 to 30 feet.

ΔE_{max} is taken as 150 millivolts for the following reason. The practice with RCN cathodically protected ships is to maintain their potentials at 860 millivolts $\pm$20 millivolts.[3, 4] The potential at which protection in sea water is obtained and which may be taken as E_{eq} is 740 millivolts.[4, 5, 6] Thus a value of 150 millivolts is reasonable for ΔE_{max}. The value of δ for sea water averaged over the yearly temperature variation in North Atlantic waters is 0.042 ohms^{-1} cm^{-1} (see Reference 7).

From experience J_{ox} for painted hulls with a deteriorated paint coating is less than 10 ma/sq ft (see Reference 3) and values of similar size have been quoted for bare steel.[5] This value will be used in this note.

Substituting the defined values in Equation 1 for the case when C = 20, 30, 40, and 60 ft respectively, one obtains values for f from which $\frac{S}{C}$ can be obtained from Wagner's graphical data. The results are tabulated in Table 1.

Discussion of Results

The values of S in the table for lower values of C are in approximate agreement with actual values except for C = 20.

For steel and magnesium anodes the width of the wooden fairing holding the steel anode and the width of rubber insulation under magnesium anodes is 16 inches to 18 inches. However in these cases the paint acts as an insulator S.

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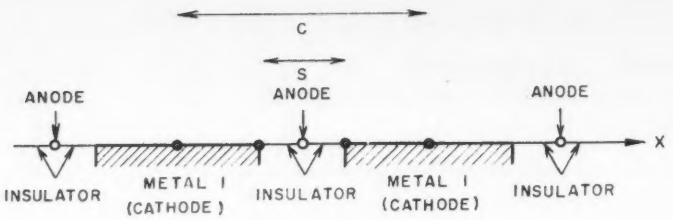

Figure 1—Wagner's model for an anode in the plane of metal to be protected.

Figure 2—Wagner's model for an anode at a distance from metal to be protected.

TABLE 1—Cathodic Protection Results

C ft	f	log f	$\frac{S}{C}$	S ft
20	0.93	—.0315	.034	.78
30	0.63	—.2007	.096	2.8
40	0.472	—.3279	.156	6.2
60	0.31	—.5086	.26	15.6

Experience has shown that with steel and magnesium anodes a vinyl patch extending 3 feet around the anodes is necessary for cathodic protection at reasonable overall total currents (i.e., in order to obtain sufficient current spread.) If a high quality alkali resistant paint is not used around the anodes, then an array on the bilge keels will protect the entire hull only as long as the paint is intact. In the case of a small tug with S = 6 inches and C = 15 feet, protection was not obtained when paint stripped around the anode. In this case the anode width was comparable to S. With the standard 10 inch wide anode, S is 16 inches and again the anode width is comparable to S. Breakdown of the vinyl film around the anodes, when it does occur for such systems, is usually confined to an area within one to two feet of the anode surface. This suggests that the S values obtained when C is 20 ft to 30 ft are of the right order.

Steel anodes on a tug where C was just under 20 ft, S about 18 inches and the anode 5 inches wide protected the hull at reasonable current outputs after the paint around the steel anodes had deteriorated.

Practical experience has shown that a current shield of 7 ft minimum size is required if protection is to be obtained on all the various sizes of vessels fitted with impressed current permanent anodes. In these cases with inert anodes, the anodes do not run the full length of the bilge keel and the working face is smaller than for steel or magnesium. Hence a higher voltage may be impressed across a normal paint film and the seven foot dimension is probably the distance over which the applied anode voltage drops to a low value so that ΔE_{max} is within the defined limits. It also has been found that the current shields around anodes do not have to be increased beyond 7 or 8 ft maximum for protection regardless of the size of C. C is given as $\frac{\Delta E_{max}\,\delta}{J_{ox}\,f}$. Wagner's graph of the function f for the model in Figure 1 begins to decrease more rapidly in the region of $\frac{S}{C} = 0.13$. From the above discussion a maximum shielding distance of 7 ft would lead to an average value of $\frac{S}{C}$ of 0.2 for most ships. Although f decreases more rapidly for values of $\frac{S}{C} > 0.13$, the decrease is not sufficiently rapid to cause an S value of 7 ft to be valid for all values of C.

When S = 7 ft, the anodes can also be considered as acting at a distance from the hull as shown in the model in Figure 2. In this case the function f begins to decrease very rapidly at $\frac{d}{c} = 0.2$. Thus this model is in better agreement with the observed fact that S need not exceed 7 ft. Using the model from Figure 1 and the same values for $J_{ox}\,\delta$, and for C and ΔE_{max}, a value of $\frac{d}{c}$ can be obtained using Wagner's graphical information. This value is .04 when C is 30 ft so that d = 1.2 ft. This value is low although practical experience has shown that 2 ft was sufficient for a partially bare tug[7] where C was of the order of 20 ft.

The effect of a decrease in conductivity can be seen in the experience of a group of destroyers with steel anodes whose cruise included a change from the Atlantic to the Baltic, a change of δ from 0.042 to .010.[8]

At the lower conductivity value, the value of f required for a C of approximately 40 ft is f = .375. From Wagner's graphs an S value of 8.5 ft would be required for protection under these conditions. In practice under the conditions described above, data are available only for magnesium and steel systems and in these cases in fresh water the paint barrier in the immediate vicinity of the anodes broke down. This indicated that in these areas ΔE_{max} must have been very much greater than 150 mv. With the paint stripped near the anodes, complete cathodic protection was impossible because S was then effectively 18 inches, the width of the wooden fairing. On returning to ocean water again, protection (i.e., polarization to 860 ±20 mv) was possible only at a greatly increased total current output. For steel impressed current systems this involved an increase from 50 to 60 amps to 150 amperes. Under these conditions ΔE_{max} is exceeded at certain portions of the hull and considerable current must be used for hydrogen reduction.

Conclusions

Wagner's analysis of the conditions for cathodic protection is applied to actual operating data and it is seen that it applies fairly well for cathodes where C is under 40 ft. Practical experience has shown that once S = about 7 feet, C can be increased very greatly in sea water and protection can still be obtained. This is due to the fact that an anode at a distance greater than 7 feet from the cathode it is protecting, acts as a point source in an infinite conducting medium. In other words δ is effectively increased with the result that f increases. As $\frac{S}{C}$ decreases with increasing f the situation described above occurs.

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Corrosion Resistance of Titanium and Zirconium In Chemical Plant Exposures*

By P. J. GEGNER and W. L. WILSON

Abstract

Corrosion tests were conducted with titanium, zirconium and a number of other metals in a wide variety of chemical plant exposures. Some of the tests were carried out under similar conditions in as many as five different plants. Titanium was found to possess low or, in many cases, nil corrosion rates in such varied exposures as wet chlorine gas, hypochlorous acid, sodium and calcium hypochlorites, sodium and potassium chlorides, sea water and a number of solutions containing wet chlorine. It was also found to be very resistant to many concentrations of calcium chloride, sodium and potassium hydroxides, nitric acid and chlorine-saturated sulfuric acid.

Zirconium exhibited low corrosion rates in nitric acid, sodium and potassium hydroxides, hydrochloric acid, dry chlorine, sodium hypochlorite, sodium and potassium chlorides, sea water and some concentrations of calcium chloride and chlorine-saturated sulfuric acid.

Results of 116 exposures involving over 800 corrosion rates on 40 different materials are described. 6.3.15

Introduction

TITANIUM AND zirconium, as measured in terms of most other metals, are new materials in the chemical industry's arsenal of weapons for combatting corrosion. Despite their relative newness, they have been known for some time to possess interesting corrosion-resisting properties. Until recently, however, both materials were available for commercial use in such limited quantities, and were so high priced in relation to all but the noble metals that they aroused comparatively little interest.

Events of the past year or so have altered this picture. Just as the titanium industry achieved an all time high in production capacity, a cut-back in the military aircraft program went into effect. As a result, titanium became readily available for non-military applications. This availability, along with a series of price reductions, has greatly stimulated the interest of the chemical industry in strong, light-weight, corrosion-resistant titanium.

Much the same story can be told of zirconium. Available for the first time in quantities in excess of those required for nuclear reactors, it is making its bid for a place in the commercial market primarily on the basis of its unusual corrosion resistance, especially to hydrochloric acid.[1, 2, 3, 4]

Most of the corrosion data on titanium and zirconium are based on laboratory tests. In many cases, data do not compare the corrosion resistance of titanium and zirconium with those materials commonly used in the specific environment. A need was recognized for supplementing the existing corrosion data on titanium and zirconium with some obtained in chemical plant exposures, at the same time comparing these two metals to some of the more commonly used ones.

A comprehensive program was set up to achieve these aims. Commercially pure titanium and zirconium were evaluated along with a number of other metals and alloys in a wide variety of corrosive chemical plant exposures. Seven plants, most of which fall in the chlor-alkali group, participated in the project.

This paper describes the experimental procedure and discusses the results of the first of the completed tests.

Experimental

Selection of Specimens

The selection of specimens for the program was of fundamental importance. Among the questions which were considered were whether to include alloys of titanium and zirconium, stressed specimens, welded specimens and galvanic couples. The final decision to use only commercially pure titanium and zirconium and standard disc-type specimens was based on generally known properties of the two materials.

Titanium has been alloyed successfully, either individually or in combinations of two or more, with manganese, aluminum, vanadium, chromium, tin, iron and molybdenum. However, these alloys have been aimed almost entirely toward improving the physical properties and/or the workability of titanium. A number of investigators[5, 6, 7, 8] have shown the corrosion properties of titanium and its alloys to be quite similar. More often than not, the addition of alloying elements has to some extent decreased the corrosion properties of titanium.

Only three zirconium alloys have been used to any extent, all in nuclear reactor applications. They are low alloys of hafnium-free, high purity zirconium containing small amounts of tin, iron, chromium and nickel. These additions improve zirconium's strength at elevated temperatures and its corrosion resistance to high temperature water and to liquid metals, while maintaining low neutron absorption characteristics.[9]

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Hafnium is normally associated with zirconium, usually up to 2 percent or so. The two metals are difficult and costly to separate. Since hafnium reportedly does not harm the corrosion resistance of zirconium in general, the only grade practical for use in the chemical industry contains hafnium to the extent of 1.5-2.2 percent.[3, 4, 9]

The use of stressed specimens of titanium and zirconium to determine stress cracking tendencies was ruled out on the basis of reports that neither material is subject to this type of failure in most chemical environments.[10, 11, 12] Keifer and Harple[10] point out that titanium could not be made to crack in 23 different types of solutions, although it did stress crack in red fuming nitric acid. Some of the solutions tested by those authors included boiling saturated sodium chloride, boiling 20 percent solutions of sodium

TABLE 1—Compositions of Titanium and Zirconium

Element	METAL EXAMINED		
	Commercial Titanium A-55	Electro-Refined Titanium	Commercial Zirconium
Fe	0.18	<0.005	0.071
Cr	....	<0.005	0.011
Mg	....	<0.001	0.014
Na	....	0.02	.....
Si	0.075	0.01	0.0035
Mn	0.068	0.01	0.0030
Al	....	<0.03	0.0052
N_2	0.006	<0.001	0.0030
O_2	0.10	0.012	0.080
H_2	0.010	0.006	.....
C	0.048	0.008	0.0059
Cl_2	....	0.04	0.020
V	....	<0.005	<0.0002
Cu	....	<0.006	0.0008
BHN	....	68	143

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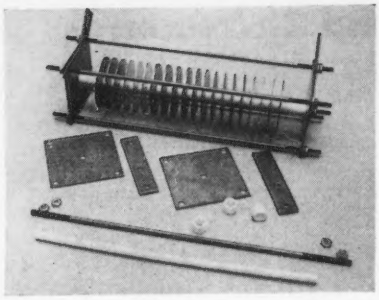

Figure 1—Spool type specimen holder.

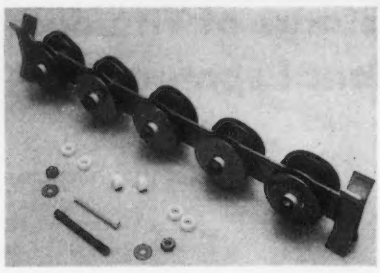

Figure 2—Pipe line type specimen holder.

chloride at pH as low as 0.3, boiling 20 percent solutions of sodium chloride containing 1 percent of various metallic salts, 20 percent ferric chloride, 42 percent boiling magnesium chloride, 65 percent boiling nitric acid, 10 percent sodium hydroxide, aqua regia and 90 percent white fuming nitric acid.

Welded specimens were not included because here again available information indicated that titanium and zirconium welds, properly made, are not subject to local attack either in the weld or in the adjacent, heat-affected zones.[12, 13] Tests of titanium welds in one experiment indicated that increasing the carbon content of titanium welds in the range of 0.015 to 0.800 percent does not adversely affect corrosion properties.[14]

Galvanic couples of titanium and zirconium with other metals were considered and ruled out on the basis that their behavior in such couples has been well established. Titanium passivates in a number of environments and exhibits a noble electrode potential similar to that of a passive 18-8 stainless steel or Monel.[6, 11, 15, 16]

Cox[17] reports that zirconium is resistant to galvanic attack when in contact with such dissimilar metals as magnesium, aluminum, zinc, lead, tin, copper, Monel, nickel, low alloy steel and stainless steels, in solutions which included synthetic sea water, tap water, 5 percent ammonium sulfate, 5½ percent sodium chloride, sodium hydroxide solution and 0.1N hydrochloric acid.

The compositions of the commercial grades of titanium and zirconium used in the program are shown in Table 1. Also shown is the composition of a high purity, electrorefined titanium used in a few of the exposures to determine if an appreciable difference existed in corrosion properties between the high purity and the commercial quality titanium materials.

All other metals and alloys were of standard, commercial quality. For each specific environment, materials were chosen which were known to be generally useful or which were desired for comparison purposes. Each plant cooperating in the program was free to add to the basic lists any metals in which it had a particular interest.

Naturally it was impractical to evaluate all the metals which might be useful in each environment. For the purpose of comparing the corrosion properties of titanium and zirconium with some of the more commonly used metals, the choices are reasonably representative.

Selection of Environments

The selection of environments in which to expose the specimens was limited necessarily to those available in the manufacturing operations in the types of plants involved. Again, a basic list of environments was chosen on the basis of general interest and each plant was free to add exposures in which it had a specific interest.

Where similar conditions existed in two or more plants, exposures were made in each such condition. This permitted a degree of comparison seldom possible in plant exposure tests.

Duration of Tests

All tests were to continue for at least one month except in the most severe exposures where it was felt too many specimens might be completely corroded away in that time. Wherever possible, periodic inspections were made of materials under test and tests were halted whenever it appeared excessive attack had taken place on a number of the coupons. In many cases, equipment outages determined the length of an exposure.

Test Procedure

Most of the exposures were made with standard Inco-type coupons, mounted, in

TABLE 2—Exposures in Calcium Chloride Solutions

Exp. No.	Corrosion Rates, Mils Per Year										
	Ti[1]	Ti[2]	Ti[3]	Ti[15]	Zr	Ni	Monel	Inconel	Nionel	Ta	Mild Steel
1	0.1	....	....	....	0.001	3	1	0.4[10]	0.06[9]	....	21[7]
2	Nil	....	....	....	0.03	8	9	8	....	....	25
3	0.001	....	....	....	0.03	23	12	7[10]	....	....	38
4[14]	Nil	....	....	....	0.4	17	11	30	29	....	52
5	0.02	....	....	....	.....	13[6]	10[6]	4[6]	....	....	55
6	Nil	....	....	....	0.009	> 37[4]	28	>30[4]	3[6]	....	> 75[4]
7	.....	....	....	....	.....	66	37	....	....	Nil	....
8	> 60[5]	....	....	49[8]	8[12]	13	12	3[6]	6[7]	....	49
	0.002	....	....	....	0.1	....	....	....	....	....	....
9	> 41[5]	....	....	....	0.1[11]	16	9	4[8]	8[8]	....	73
	Nil	....	....	....	.....	....	....	....	....	....	....
10	0.002[13]	32	5[12]	....	.....	....	....	....	....	....	....
	.....	9[11]	0.01	....	.....	....	....	....	....	....	....
11	31[8]	....	....	....	> 70[4]	13	12	2[6]	....	....	103
12	22[8]	....	....	....	>118[4]	16	11	3[6]	1[7]	....	114

[1] Test specimens from Source No. 1. This was the source of the specimens used throughout this test program.
[2] Test specimens from Source No. 2.
[3] Test Specimens from Source No. 3.
[4] Specimens were completely consumed during the test.
[5] One of two duplicate specimens was completely consumed.
[6] Slight or shallow pitting attack.
[7] Moderate pitting attack.
[8] Severe pitting attack.
[9] Slight attack under spacer.
[10] Moderate attack under spacer.
[11] Severe attack under spacer.
[12] One specimen was severely attacked while the duplicate specimen was only slightly attacked.
[13] Average of four specimens.
[14] This test spool was in sludge at the bottom of a tank for about one half of the exposure time.
[15] Electro-refined, high-purity titanium.

Exposure No.	Environment	Temperature	Test Duration, Days
1	40% $CaCl_2$ with suspended NaCl and $CaSO_4$	230 F	169
2	55% $CaCl_2$ with suspended NaCl and $CaSO_4$	220 F	174
3	55% $CaCl_2$ with suspended NaCl and $CaSO_4$	235 F	120
4	55% $CaCl_2$ Saturated with NaCl and $CaSO_4$	235 F	84
5	62% $CaCl_2$	310 F	30
6	62% $CaCl_2$	310 F	188
7	62% $CaCl_2$	310 F	61
8	62% $CaCl_2$	310 F	97
9	62% $CaCl_2$	310 F	146
10	62% $CaCl_2$	310 F	98
11	73% $CaCl_2$	350 F	83
12	73% $CaCl_2$	350 F	49

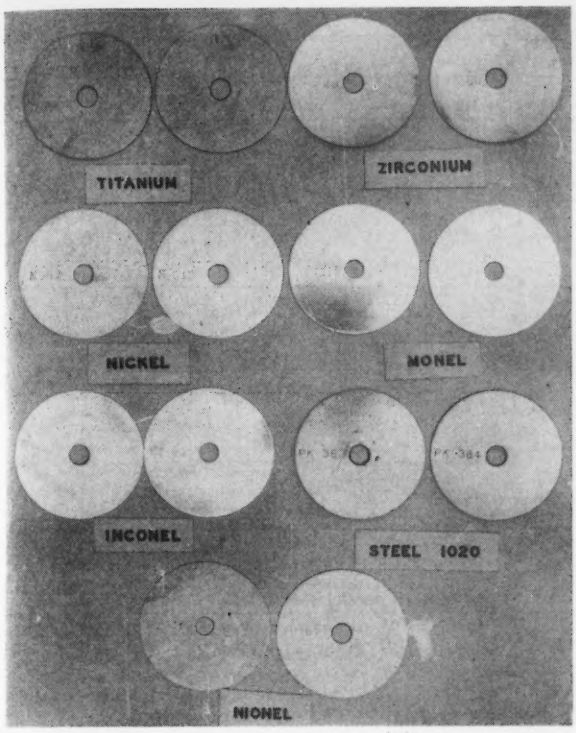

Figure 3—Typical set of specimens before exposure.

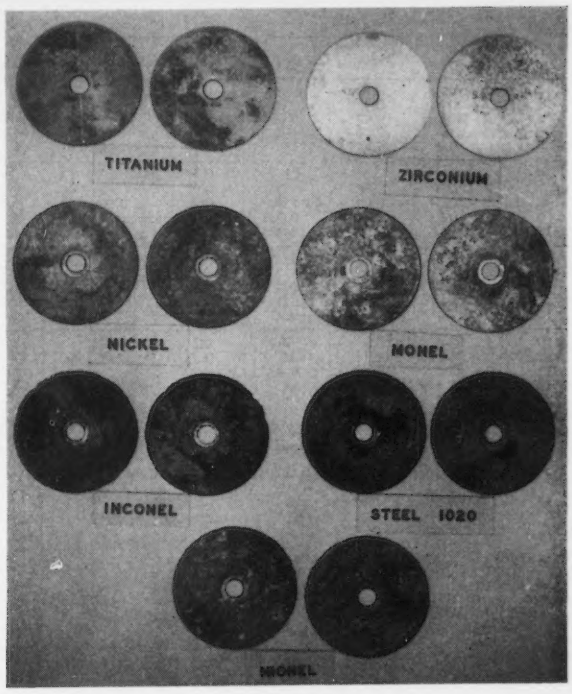

Figure 4—Typical set of specimens after 85 days exposure to 55 percent $CaCl_2$. Specimens pictured are same as shown in Figure 3.

duplicate, on one of two types of specimen holders[18] (see Figures 1 and 2). These holders provide for the complete isolation of the coupons from one another and from the holder itself. The spool type is standard for installation in most process vessels where adequately sized openings and space are available. The pipe-line type may be installed in pipe lines 3 inches or larger, with standard size coupons, with a minimum interference to flow through the line.

Specimen holders were made of titanium, Monel, stainless 316, steel or Teflon to suit the specific exposure. Insulating sleeves and spacers were made of Teflon.

All specimens were cleaned, solvent washed, dried, weighed and photographed prior to exposure. A typical photograph of a set of specimens for one exposure is shown in Figure 3. Upon completion of the test, the coupons were cleaned by whatever method was called for by the condition of the specimen (i.e., electrolytic cleaning, scrubbing lightly with scouring powder, etc.). After they were cleaned and reweighed, the specimens were again photographed, as shown in Figure 4.

Reporting of Results

In reporting the test results, a number of nil or extremely low corrosion rates were observed. A common practice is to report such rates as "less than one mil per year (< 1 mpy)." Since most of the tests ran for 30 days or more, these nil and very low rates were felt to be significant. The nil rates are therefore reported as such and rates of less than one mil per year are calculated to the nearest significant figure.

In most cases, pit depths were measured and recorded. However for the sake of conciseness, pitting is described in the tables in general terms.

Slight or shallow pitting is usually less than about 3 mils, moderate pitting may run 3 to 10 mils, severe pitting is over 10 mils, all in terms of length of exposure. Spacer or crevice attack, edge attack and other unusual surface changes are also noted, again in broad terms.

Discussion

Calcium Chloride

The results of exposures in calcium chloride solutions are shown in Table 2. Titanium and zirconium exhibited extremely low corrosion rates in 40 percent and in 55 percent solutions, both performed erratically in 62 percent solutions, and failed consistently in 73 percent solutions.

In the 40 percent and 55 percent solutions at temperatures up to 235 F, titanium showed nil or negligible rates, even in the presence of suspended salt and calcium sulfate, at velocities up to five feet per second.

In 62 percent calcium chloride at 310 F, titanium showed an entirely unexpected variation in its resistance. In two cases, Exposures 8 and 9, one specimen on a spool was unattacked while its duplicate was completely consumed. Exposure 10 shows the results obtained with titanium specimens from three different sources. In this exposure, four of the regular specimens were virtually unaffected, while specimens from the other two sources showed the wide variation in corrosion rates. This test indicated that the regular specimens were not unusual in showing great extremes in resistance to 62 percent calcium chloride.

Apparently this is a borderline condition for maintaining the passivity of titanium. As long as the surface is passive, corrosion resistance to this solution is nearly complete. A breakdown of passivity, for any of a number of possible reasons, allows titanium to corrode at a very rapid rate. Observations made during these exposures indicated that passivity failed in local areas, often at the machined edges of the specimens. These observations also revealed there was an induction period, varying from several days to several weeks, before the passive film broke down.

Both specimens of the electrorefined, high purity titanium suffered severe pitting attack in Exposure 8. Apparently the permanence of titanium's passivity in 62 percent calcium chloride is not dependent upon its purity.

In 73 percent calcium chloride at 350 F, titanium showed severe pitting and a high rate of attack on all four specimens used in Exposures 11 and 12.

The results obtained in the higher concentrations of calcium chloride are not in accordance with some previously published data.[19] In a ten day test in 70 percent calcium chloride at 318 F, a nil corrosion rate was reported. In 80 percent calcium chloride at 392 F, the corrosion rate of titanium was reported to be only 0.2 mils per year.

It was suspected that possibly some

TABLE 3—Exposures in Alkali Solutions

Exp. No.	Corrosion Rates, Mils Per Year												
	Ti	Zr	Ni	Monel	Inconel	Hast. C	304	316	Cb.	Ampco 8	Mild Steel	Cast Iron	Croloy 9
1	0.04	0.2[4]	0.2	0.3	0.05	....	....	....	....	....	4[4]	....	....
2	>30[1]	0.4	....	....	....	0.5[7]	1[7]	....	....	....	5	....	3
3	Nil	0.07	0.003	Nil	Nil	....	....	....	....	....	0.6[7]	....	....
4	Nil	0.09	Nil	Nil	Nil	....	....	....	....	....	0.5[7]	....	....
5	0.9	0.2	1[4]	0.9[4]	....	....	....	3	....	....	8	....	....
6	0	0.07	0.2[6]	0.7[6]	0.3[6]	....	....	....	....	4	9	....	....
7	0.5	0.08	0.02	0.02	0.02	....	....	....	....	1	5	....	....
8	0.01	0.09	0.009	0.02	0.008	....	....	....	....	....	0.7	....	....
9	0.02	0.06	0.007	0.007	....	....	....	....	....	....	0.2	....	....
10	0.06	0.06	0.003	0.003	Nil	....	....	....	....	....	1	....	....
11	0.4	0.06	0.003	0.002	Nil	....	....	....	....	....	0.05	....	....
12	>36[1]	3	18	17	....	....	....	....	....	....	>95[1]	....	....
13	Nil	0.05	Nil	Nil	Nil	....	....	....	....	....	0.6[6,8]	....	....
14	1	0.9	0.05[4]	0.2	0.09	....	....	....	....	2	17	....	....
15	0.6	0.5	0.02	0.05	0.03	....	....	....	....	....	2	....	....
16	2	0.8	0.02	0.1	0.008	....	....	....	....	0.9	39	....	....
17	5	1	0.3	0.7	0.4	....	....	....	....	....	>77[1]	....	....
18	1	0.8	0.002	0.005	....	....	....	....	....	....	2	....	....
19	7	2	1	0.9	1	....	....	....	0.8	....	>80[1]	....	....
20	—[2]	24	—[2]	78[3]	77[5]	....	....	....	....	....	37	—[2]	....

[1] Specimens were completely consumed during the test.

[2] Specimens were lost due to failure of the suspending wire.

[3] One of two duplicate specimens was lost.

[4] Slight pitting attack.

[5] Slight pitting attack on one of two specimens.

[6] Moderate pitting attack.

[7] Slight attack under spacer.

[8] Severe attack under spacer.

Exposure No.	Environment	Temperture	Duration of Test, Days
1	5-10% NaOH	70 F	124
2	7% NaOH solution containing 53% NaCl. 7% $NaClO_3$ and 80-100 ppm of NH_3	375 F	198
3	9-11% NaOH containing about 15% NaCl.	180 F	207
4	13% KOH containing about 13% KCl	85 F	207
5	50% NaOH containing a small amount of free Cl_2	100 F	153
6	50% NaOH containing about 750 ppm of free Cl_2	200 F	161 (Vessel cathodically protected)
7	50% NaOH	135 F	135
8	50% NaOH	100 F	162
9	50% NaOH	105 F	171
10	50% NaOH	100 F	207
11	50% KOH	80 F	207
12	52% NaOH, 16% NH_3	280 F	175
13	0.6% NaOH, 2% $NaClO_3$, Trace of NH_3.	265 F	175
14	73% NaOH	240 F	81
15	73% NaOH	240 F	162
16	73% NaOH	230 F	126
17	73% NaOH	240 F	183
18	73% NaOH	235 F	171
19	73% NaOH	265 F	200
20	Caustic fusion, 73% to anhydrous NaOH	250 to 1000 F	14

minor impurity was responsible for the poor results obtained with titanium in the higher concentrations of calcium chloride. A laboratory test was conducted in which titanium specimens from two sources were exposed in production-quality calcium chloride and in chemically-pure calcium chloride, each at 73 percent concentration and at 375 F. After 35 days, some specimens in each solution showed characteristic pitting. This indicates that the breakdown of titanium's passivity is not due to impurities in calcium chloride but is a function of concentration and/or temperature.

Zirconium, like titanium, offers outstanding resistance to calcium chloride at 40 percent and 55 percent concentrations, even in the presence of suspended salt and calcium sulfate. In 62 percent solutions, less erratic results were obtained than with titanium. There was, however, a considerable difference in the two specimens in Exposure 8, and both specimens exhibited severe spacer attack in Exposure 9. In 73 percent solutions, zirconium specimens were completely consumed in all cases, showing a very high rate of attack.

Tantalum showed a nil corrosion rate in 62 percent solution. Other than this, no other material was entirely satisfactory in the higher concentrations. Monel, by virtue of its freedom from pitting, may be considered more useful than materials such as Inconel and Nionel which possess lower corrosion rates but suffer moderate to severe pitting. This has been borne out in actual plant practice.

Alkali Solutions

Both titanium and zirconium demonstrated excellent resistance to alkali solutions over a wide range of concentrations and temperatures (Table 3).

Titanium was almost completely resistant to potassium hydroxide at 13 percent and at 50 percent concentrations. In sodium hydroxide, it was very resistant to solutions ranging from 10 percent at 180 F to 73 percent at 240 F. In 50 percent sodium hydroxide, the rates were less than one mil per year at temperatures up to 135 F. At 50 percent concentration and 200 F (Exposure 6), the rate was up to 3 mils. In this exposure, the presence of a small amount of chlorine apparently was less an influence than the higher temperature. In 73 percent sodium hydroxide, corrosion rates in six exposures varied from less than a mil to 7 mils per year. This variation probably is related to differences in temperature since the 7 mil rate (Exposure 18) is at the highest temperature. There is a good correlation generally between the corrosion rates of titanium and the other materials, particularly steel, in these various 73 percent exposures.

In Exposure 2, titanium failed in 7 percent sodium hydroxide at 375 F, in the presence of a small amount of ammonia. In Exposure 12, it failed in 52 percent sodium hydroxide at 280 F, in the presence of 16 percent ammonia. These failures may have been due to the high temperatures and/or an effect by the ammonia.

In caustic fusion (Exposure 20), the titanium specimens were lost but a high corrosion rate would be expected by virtue of the fact the titanium wire from which the specimens were suspended was eaten through in a matter of a few days.

The performance of titanium in strong alkali solutions came as somewhat of a surprise, probably because lack of information had created the impression it was not satisfactory in concentrations above 40-50 percent.

Zirconium was consistently good in all alkali solutions. In Exposure 12, in the presence of ammonia, zirconium was the only material with a low corrosion rate. In caustic fusion, the attack was higher than was anticipated on the basis of previously reported data.[4,20] The addition of sulfur to the fused caustic soda to precipitate iron may account for the relatively high corrosion rate.

Mild steel showed low corrosion rates in both potassium and sodium hydroxides up to 50 percent. The rates were affected in Exposures 5 and 6 by the presence of free chlorine and in Exposure 7 by a higher temperature.

Nickel, Monel and Inconel were generally excellent. The slight to moderate pitting reported in Exposures 5 and 6 is probably due to the presence of chlorine.

TABLE 4—Exposures in Gaseous Chlorine

Exp. No.	Corrosion Rates, Mils Per Year								
	Ti	High-Purity Ti	Zr	Monel	Hastelloy C	Chlorimet 3	SS 316	Mild Steel	Columbium
1	–[11]	.....	0.02	0.3[13]	0.001	0.006	0.2	0.2	.....
2	–[12]	.....	–[12]	0.2	0.02	0.01	–[12]	13[3]	.....
3	Nil	.....	20	.....	0.07	0.5[7]	....	....	.....
4	Nil	.....	> 80[1]	>80[1]	0.07	0.4	....	....	.....
5	Nil	.....	20	.....	0.05	0.6	....	....	.....
6	Nil	.....	> 90[1]	....	0.8	11	....	....	.....
7	Nil	.....	> 43[1]	....	0.9[7]	9[7]	....	....	.....
8	0.02	.....	50[5]	....	6[8]	20[8]	....	....	.....
9	0.008	.....	> 30[1]	....	5[6]	54[6]	....	....	.....
10	0.03	.....	> 90[1]	....	0.7[7]	8[9]	....	....	.....
11	0.01	.....	73[6]	....	17[8]	6[8]	....	....	.....
12	Nil	.....	> 30[1]	....	2[6]	31[6]	....	....	.....
13	0.01	.....	56[6]	....	210[6]	149[5,9]	....	....	.....
14	0.007	.....	> 80[1]	....	5[5]	3[5]	....	....	.....
15	0.1	.....	> 60[1]	>60[1]	1[9]	12[9]	....	....	.....
16	0.03	.....	44	>60[1]	1[9]	13[9]	....	....	.....
17	Nil	.....	>150[2] 51[6,10]	186[4]	222[5]	271[6]	....	....	.....
18	0.0008	0.01	18	....	23	53[5]	....	....	Very low[14]

[1] Specimens were completely consumed during the test.
[2] One of two duplicate specimens was completely consumed.
[3] Slight or shallow pitting attack.
[4] Moderate pitting attack.
[5] Severe pitting attack.
[6] Specimens were perforated by very severe pitting attack.
[7] Slight attack under spacer.
[8] Moderate attack under spacer.
[9] Severe attack under spacer.
[10] Severe edge attack.
[11] The titanium specimens apparently ignited upon exposure.
[12] Zirconium specimens were destroyed and stainless steel 316 specimen was damaged when the titanium specimens ignited.
[13] One Monel specimen was damaged when the titanium specimens ignited.
[14] Original weights were lost. Duplicate specimens were not visibly attacked during 203 days.

Exposure No.	Environment	Temperature	Duration of Test, Days
1	Dry Cl_2 gas	0 F	139
2	Dry Cl_2 gas	140 F	36
3	Wet Cl_2 gas	50 F	139
4	Wet Cl_2 gas	60 F	73
5	Wet Cl_2 gas	100 F	133
6	Wet Cl_2 gas	170 F	67
7	Wet Cl_2 gas	180 F	137
8	Wet Cl_2 gas	190 F	18
9	Wet Cl_2 gas	190 F	202
10	Wet Cl_2 gas with some condensed water and organic solids present	170 F	67
11	Wet Cl_2 gas with some condensed water and organic solids present	190 F	18
12	Wet Cl_2 gas with some condensed water and organic solids present	190 F	203
13	Wet Cl_2 gas containing salt brine spray	190 F	28
14	Wet Cl_2 gas containing salt brine spray	190 F	74
15	Wet Cl_2 gas	205 F	99
16	Wet Cl_2 gas with some condensed water and organic solids present	205 F	98
17	Wet Cl_2 gas containing salt brine spray	205 F	40
18	Wet Cl_2 gas containing salt brine spray	205 F	203

Ampco 8, an aluminum bronze, showed useful resistance in both 50 percent and 73 percent solutions. Columbium was excellent in 73 percent sodium hydroxide at 265 F.

Gaseous Chlorine

In wet chlorine gas, titanium was found to possess an unusually high degree of resistance, but in dry chlorine gas, it failed catastrophically, by ignition (Table 4). Zirconium, on the other hand, was very resistant to the dry gas, but was severely corroded in wet chlorine.

These tests were conducted in most of the various chlorine gas exposures to be found in typical electrolytic chlorine plants. Spools were exposed in the vapor space above the anolyte in chlorine cells, in the gas stream in collection headers, partly in the gas and partly in the condensed water and organic sludge which accumulates in collection headers, in various sections of gas coolers, and in dry gas.

In none of 15 exposures to wet gas, over a range of temperatures from 50 F to 205 F, did titanium exhibit a significant corrosion rate. In about half of these exposures, the corrosion rate was nil. In several dry gas exposures, the titanium failed almost immediately, apparently by ignition.

The dividing line between wet and dry chlorine as far as corrosion resistance of titanium is concerned has been reported to be as low as 50 ppm water.[11]

Zirconium was unsatisfactory in all wet gas exposures, regardless of temperature. Many of the specimens were consumed during the test periods. Those which were recovered almost invariably were severely pitted. In dry chlorine, the attack on zirconium was negligible.

Hastelloy C proved very resistant to wet chlorine up to 100 F and, except for slight spacer attack, was not attacked appreciably at 180 F. Chlorimet 3 showed good resistance to wet chlorine up to 100 F.

While an exact corrosion rate for columbium could not be reported because the record of the original weights was lost, it was obvious upon inspection of the specimens that this material had suffered little or no attack in wet chlorine gas at 205 F (Exposure 18).

TABLE 5—Exposures in Solutions Containing Free Chlorine

Exposure No.	Corrosion Rates, Mils Per Year				
	Titanium	Zirconium	Monel	Hastelloy C	Chlorimet 3
1	1	46[6]	....	32[6]	38[6]
2	0.03	94[3]	....	3	14
3	0.4	> 90[1]	98	3[5]	26[5]
4	0.02	29[3]	....	44[5]	156[5]
5	Nil	>150[2] 71[6,8]	158[8]	155[8]	186[3] .... —[6,7]
6	0.002	—[6,7]	....	—[5,7]	
7	Nil	> 29[1]	....	25[8]	97[8]

Exposure No.	Environment	Temperature	Duration of Test, Days
1	1% HCl saturated with Cl_2, CO, and CO_2,	80 F	22
2	River water saturated with Cl_2	200 F	14
3	Sea water saturated with Cl_2,	203 F	63
4	About 20% NaCl saturated with Cl_2 (cell anolyte)	190 F	28
5	About 20% NaCl saturated with Cl_2 (cell anolyte)	205 F	40
6	About 20% NaCl saturated with Cl_2 (cell anolyte)	195 F	46
7	About 20% NaCl saturated with Cl_2 (cell anolyte)	200 F	204

[1] Specimens were completely consumed during the test.
[2] One of two duplicate specimens was consumed.
[3] Severe pitting attack.
[4] Very slight attack under spacer on one specimen only.
[5] Severe attack under spacer.
[6] Severe edge attack.
[7] Over-all rates were not calculated, localized attack was very severe.
[8] Specimens perforated by very severe pitting attack.

TABLE 6—Hypochlorite Exposures

Exp. No.	Corrosion Rates, Mils Per Year									
	Ti	High-Purity Ti	Zr	Hast. C	Chlorimet 3	316	Worthite	Duriron	Durichlor	Mild Steel
1	0.001	.....	0.3[4]	0.6	19	....	....	....	....	....[2]
2	Nil	.....	12.[4]	0.5	3	22.[4]	....	....	....	....
3	Nil	.....	1[4]	0.01	1[4]	102.[4]	172.[4]	....	....	....
4	0.1	.....	0.9[4]	0.05	0.3[3]	2[5]	0.3[4]	....	....	....
5	0.001	Nil	2[4]	9	40	....	....	....	....	....
6	0.01	.....	0.07	0.1	0.3	....	....	....	0.8	....
7	0.1	.....	4[2]	46[2]	....	>100[1]	....	12[2]	7[2]	>200[1]

[1] Specimens were completely consumed during the test.

[2] Severe pitting attack.

[3] Slight attack under spacer.

[4] Severe attack under spacer.

[5] Perforated under spacer.

Exposure No.	Environment	Temperature	Duration of Test, Days
1	Up to 15% Cl_2 with some HOCl, Cl_2 and H_2O, gas exposure	110 F	204
2	Air containing Ca $(OCl)_2$, Cl_2 and H_2O, gas exposure	180 F	204
3	18-20% solution of $Ca(OCl)_2$ liquid exposure	70-75 F	204
4	Mixture containing $Ca(OH)_2$, $Ca(OCl)_2$ and HOCl	75 F	42
5	17% HOCl solution and Cl_2O and Cl_2 gases, intermittent gas-liquid exposure	100 F	203
6	Up to 16% NaOCl solution containing NaOH, liquid exposure	60 F	170
7	Solution containing 1.5-4% NaOCl, 12-15% NaCl and 1% NaOH liquid exposure	150-200	

With the exception of titanium, all materials tested were good in dry chlorine, although the rate for steel in Exposure 2 is somewhat high. This corrosion rate probably was affected by the ignition of the titanium specimens on the same test spool.

Solutions Containing Free Chlorine

Titanium proved to be extremely resistant to attack in chlorine-saturated water, sea water, brine and weak hydrochloric acid, while zirconium was severely attacked in these solutions (Table 5).

In the absence of chlorine, all the materials involved would be expected to be resistant to these solutions. But titanium was the only material which successfully resisted attack in the chlorine-saturated solutions.

Zirconium demonstrated again its complete lack of resistance to exposures in which wet chlorine is present.

With the exception of Exposure 1, the rate of attack on Hastelloy C and Chlorimet 3, which normally would be expected to be usefully resistant in these solutions, was greatly accelerated by the high temperatures involved.

Hypochlorite Exposures

Titanium suffered little or no attack in a variety of hypochlorite exposures, while zirconium was satisfactory only in 16 percent sodium hypochlorite solution (Table 6).

Titanium demonstrated once again its remarkable resistance to solutions related to wet chlorine in their action on metals. It was completely resistant, or nearly so, to moist chlorine monoxide (Cl_2O), to calcium hypochlorite ($CaOCl_2$), to hypochlorous acid (HOCl), and to sodium hypochlorite (NaOCl).

In the batch manufacture of sodium hypochlorite, titanium showed negligible attack. In this operation, the initial exposure is to 20 percent sodium hydroxide which is gradually chlorinated to about 16 percent sodium hypochlorite.

Zirconium exhibited generally low corrosion rates in these hypochlorite solutions but suffered pitting and/or spacer attack in all cases except in the 16 percent sodium hypochlorite. These results are in line with zirconium's lack of resistance to wet chlorine.

Hastelloy C showed up well in chlorine monoxide, calcium hypochlorite and 16 percent sodium hypochlorite. Chlorimet 3 and Durichlor had low rates in the 16 percent sodium hypochlorite.

Sulfuric Acid Solutions

Table 7 shows the results of tests conducted in a variety of sulfuric acid solutions, most of them chlorine-saturated. In the presence of chlorine, the range of sulfuric acid concentrations in which titanium exhibited low corrosion rates was extended to as high as 72 percent, whereas zirconium suffered a loss of resistance in acid concentrations below 60 percent.

In pure sulfuric acid, titanium is not considered resistant above 5 percent at room temperature.[2,15] In Exposure 1, it proved resistant to 5 percent acid at 90 F in the presence of zirconyl sulfate. A metal ion effect may have contributed to this very low corrosion rate. In Exposure 2, titanium was resistant to 45 percent acid at 105 F in the presence of sodium dichromate, sodium sulfate and chromic sulfate. Here, a combination of oxidizing dichromate and chromate metal ions may have accounted for titanium's resistance. In Exposures 3 to 9, excepting Exposure 7 (61 percent acid), titanium showed very little attack to chlorine-saturated sulfuric acid solutions ranging from 40 percent to 72 percent concentration. The beneficial effect of chlorine is apparent in these exposures.

Zirconium, on the other hand, is considered useful in pure sulfuric acid up to 70-75 percent at temperatures up to 140 F.[2,4] In Exposures 1 and 2, the presence of zirconyl nitrate in 5 percent acid, and of sodium dichromate, sodium sulfate and chromic sulfate in 45 percent acid had no appreciable effect on the corrosion rate of zirconium.

Beginning with Exposure 3, the presence of chlorine obviously resulted in severe attack on zirconium in acid concentrations between 40 percent and 54 percent. But corrosion rates in the 61 percent to 72 percent acid range were quite low, while at 82 percent and higher concentrations excessive corrosion occurred.

An analysis of the behavior of zirconium in chlorine-saturated sulfuric acid indicates it follows a well-defined pattern. In pure sulfuric acid, Golden, Lane and Ackerman[2] found that zirconium is very resistant until an acid concentration of 80 percent is reached. At 80 percent, the rate of attack increased very sharply.

In the lower concentrations of chlorine-saturated acid, the chlorine attacks zirconium severely, just as it does in other solutions. As the concentration of the acid increases, it tends to have a dehydrating effect on the chlorine. Apparently, above 60 percent strength, the chlorine acts as if it were dry and the chlorine-saturated sulfuric acid behaves as would the pure acid. This would account for the low rates in 61 percent, 62 percent and 72 percent acid, and for the rapid corrosion in the 82 percent and stronger acid.

The dehydration of chlorine in strong sulfuric acid may also account for the erratic behavior of titanium in the 61 percent-72 percent concentration range. This range may represent a borderline condition for the passivity of titanium which renders it almost completely resistant to wet chlorine.

Chlorimet 3 was resistant throughout the entire range of these sulfuric acid exposures. Hastelloy C was generally good except in 84 percent acid. Hastelloy D and Nionel were resistant in 54 percent and in 84 percent acid.

Nitric Acid Solutions

In nitric solutions at temperatures up to boiling, titanium was virtually unaffected in 35 percent to 65 percent concentrations, while zirconium was very resistant in all concentrations (Table 8).

The solutions involved are typical of those found in the separation and concentration of nitric acid from zirconium, uranium, plutonium, thorium, hafnium and manganese raffinates. In this particular operation, the raffinate contains zirconyl nitrate, which is found in the various solutions in amounts ranging from trace to saturation.

TABLE 7—Exposures in Sulfuric Acid Solutions

Exp. No.	Corrosion Rates, Mils Per Year														
	Ti	High Purity Ti	Zr	Nionel	Hast. B	Hast. C	Hast. D	Hast. F	Chlorimet 3	304	316	Carpenter 20	Worthite	Duriron	Durichlor
1	1	...	1	Nil	44	2	...	1	....	Nil	1	1	....	....	....
2	0.03	...	0.7	....	..	....	....	....	....	..	41	....	0.1	....	....
3	0.04	...	>300[1]	....	...	1	...	...	—[8]	..	..	65[6]	....	0.5	0.4
4	0.1	...	0.6[5]	....	...	2	...	...	4	..	..	20[5]	....	0.2[7]	0.5[5]
5	0.03	0.01	19[5]	>60	...	0.5	74	...	1	..	..	> 80	....	2[5]	0.3[4]
				92	...	....	....	...	....	..	..	0.3[2]	....	....	....
6	0.08	...	23[5]	....	...	0.7	...	...	0.2	..	..	>250[1]	....	0.6[6]	0.2[6]
			0.3[3]	....	...	....	....	...	....	..	..	....	....	....	....
7	>45[2]	...	3	....	...	2[6]	...	...	1	..	..	> 70[1]	....	2[5.7]	1[5.7]
	10														
8	0.07	...	0.1	0.04[6]	...	0.4	1	...	0.3	..	..	0.6[6]	....	0.3[6]	0.1[3.6]
9	0.2	...	0.8	....	...	0.8	...	...	1	..	..	1[6]	....	1	0.9[4.6]
10	>47	...	> 45	0.02	...	0.2	0.06	...	0.05	..	..	0.2[6]	....	0.1[7]	0.04
11	—[9]	...	—[9]	....	...	>55	...	...	0.8	..	..	15	....	3[7]	0.7[4.6]
						3[2]									
12	—[9]	...	—[9]	....	...	0.3	...	...	0.2	..	..	0.08	....	23[5.7]	16

[1] Specimens were completely consumed during the test.

[2] One of two specimens was completely consumed.

[3] Slight pitting attack.

[4] Moderate pitting attack.

[5] Severe pitting attack.

[6] Slight attack under spacer.

[7] Severe attack under spacer.

[8] Specimen was above liquid level.

[9] Specimens were not exposed because preliminary tests indicated very high corrosion rates.

Exposure No.	Environment	Temperature	
1	5% H_2SO_4 with 3-4% zirconyl sulfate and saturated with air	90 F	15 day test
2	45% H_2SO_4 with 8% $Na_2Cr_2O_7$, 8% Na_2SO_4, 3% $Cr_2(SO_4)_3$	105 F	7 day laboratory test
3	40% H_2SO_4 saturated with Cl_2	Ambient	17 day test
4	45% H_2SO_4 saturated with Cl_2	Ambient	192 day test
5	45% H_2SO_4 saturated with Cl_2	Ambient	113 day test
6	54% H_2SO_4 saturated with Cl_2	Ambient	36 day test
7	61% H_2SO_4 saturated with Cl_2	Ambient	132 day test
8	62% H_2SO_4 saturated with Cl_2	Ambient	116 day test
9	72% H_2SO_4 saturated with Cl_2	Ambient	25 day test
10	82% H_2SO_4 saturated with Cl_2	Ambient	129 day test
11	84% H_2SO_4 saturated with Cl_2	Ambient	132 day test
12	96% H_2SO_4 saturated with Cl_2	Ambient	132 day test

TABLE 8—Exposures in Nitric Acid Solutions

Exp. No.	Corrosion Rates, Mils Per Year																				
	Ti	Ti[11]	Zr	Incoloy	Incoloy 804	Nionel	Hast. B	Hast. C	Hast. F	304	304L	309	310	311	316	316L	329	347	430	446	Carpenter 20	Ta	Al
1	13	8	..	...	...	...	..	....	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..
2	20	..	0.9	...	...	5	..	>600[1]	11	5	10	3	2	12	12	..	4	7	22	6	7	0.06	>690[1]
3	0.03	..	0.4	...	...	6	..	[2]	6	10	27	16	3	16	43	..	10	18	22	8	17	0.2	>320[4]
4	0.03	..	0.5	...	17	26	..	..	29	27	..	40	10	..	..	..	24	..	..	15	47	0.05	..
5	Nil	..	..	33	21	....	..	..	..	49[4]	..	60[5]	10	..	..	86[4]	..	..	..	..	..	..	..
	Nil[3]	..	..	....	....	....	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..
6	0.1	..	0.2	...	15	21	..	..	22	26	..	28	7	..	..	..	19	..	..	13	42	0.04	..
7	0.06	..	0.2	...	...	13	..	>290[1]	8	16	33[9]	29[5]	5	15	49	..	12	27	22[6]	9	29	0.01	>350[1]
8	Nil	..	0.09	...	...	26	...	>600[1]	21	63	120[4]	88	18	41	160[4]	..	32	64	60	28	49	0.1	>690[1]
9	Nil	..	0.8	...	...	23	1100	1120	29	69	131[4]	..	..	..	114[4]	..	..	..	..	..	38	..	..
10	Nil[7]	..	..	...	...	....	..	..	..	57[9]	60[3]	..	139[5]	..	..	231[3]	..	..	..	..	90[5]	..	..
										70[10]			52[8]										
													450[3]										

[1] Specimens were completely consumed during the test.

[2] Specimens were completely embrittled and disintegrated during disassembly.

[3] Welded specimens.

[4] Edge attack.

[5] Intergranular attack.

[6] Incipient pitting.

[7] Titanium was coupled to SS 310.

[8] SS 310 was coupled to titanium.

[9] Annealed specimen.

[10] Specimen as received.

[11] Electro-refined, high-purity titanium.

Exposure No.	Environment	Temperature	Duration of Test, Days
1	5-10% HNO_3, partially submerged	240 F	36
2	2-21% HNO_3, 3-13 ppm, Cl^-, 0-2.5% zirconyl nitrate, partially submerged	250 F	10
3	35-45% HNO_3, 3-20 ppm, Cl^-, 10-20% zirconyl nitrate, vapor and liquid exposure	250 F	21
4	35-45% HNO_3, saturated with zirconyl nitrate, vapor exposure	245 F	29
5	35-45% HNO_3, saturated with zirconyl nitrate, liquid exposure	245 F	34
6	35-45% HNO_3, saturated with zirconyl nitrate, liquid exposure	245 F	29
7	35-45% HNO_3, 3-20 ppm Cl^-, 10-20% zirconyl nitrate, liquid exposure	250 F	21
8	46-65% HNO_3, 5 ppm Cl^-, 11-13% zirconyl nitrate, liquid exposure	258 F	10
9	65% HNO_3, up to 15% zirconyl nitrate, liquid exposure	260 F	6
10	65% HNO_3, small amount of zirconyl nitrate, liquid exposure	265 F	36

Titanium was found to be unsatisfactory in nitric acid ranging from 2 percent to 21 percent at 240 F-250 F. In 35-45 percent acid, in vapor, liquid and interface exposures at 245 F-250 F, corrosion rates were nil or insignificant. In 46-65 percent acid and in azeotropic 65 percent acid, titanium was again almost completely resistant.

In Exposure 5, welded titanium specimens were compared with unwelded specimens. Both showed nil corrosion. In Exposure 10, titanium was coupled with stainless 310. Neither material was adversely affected by the couple.

Electrorefined, high purity titanium showed a lower corrosion rate in 5-10 percent acid than the commercially pure material. Whether the difference is significant is questionable.

Zirconium proved very resistant to all concentrations of nitric acid, under all exposure conditions. Only tantalum was as good over the entire acid range.

Of the other materials tested, stainless 310 was the only one which exhibited generally useful corrosion rates. Several

TABLE 9—Hydrogen Chloride and Hydrochloric Acid Exposures

Exp. No.	Corrosion Rates, Mils Per Year											
	Ti	Zr	Monel	Hast. B	Hast. C	Chlorimet 2	Duriron	Durichlor	Mild Steel	Cast Iron	Pt-Plated Ti	Ta
1	> 130[1]	36	0.2	....	0.05	....	41	46	27	29	....	....
2	4	1[3]	0.7	0.4	0.7[3]	0.3	....	....	....	....	....	....
3	> 57[1]	0.4[2]	....	3	30	4	....	....	....	....	....	....
4	....	3[4]	....	> 40[1]	....	90	....	....	....	....	....	....
5	17	1.3	....	....	....	....	....	....	....	....	0.2	0.003

[1] Specimens were completely consumed during the test.

[2] Slight pitting attack on edge of one of two specimens.

[3] Slight attack under spacer.

[4] Moderate attack under spacer and at edges.

Exposure No.	Environment	Temperature	Duration of Test, Days
1	Perchlorethylene-carbon tetrachloride mixture saturated with HCl and Cl_2 with about 20-30 ppm of water	250 F	47
2	Dichlorobenzene containing 4-5% hydrogen chloride	355 F	53
3	32-33% hydrochloric acid	Ambient	...
4	36% hydrochloric acid containing about 200 ppm of Cl_2	Ambient	163
5	Duplicate test in Exposure No. 4	...	17

direct comparisons of stainless 304 with stainless 304L (Exposures 2, 3 and 7) indicate that low carbon stainless steels are less resistant to nitric acid solutions than the standard materials.

The generally poor performance of stainless steels came as somewhat of a surprise in view of the widespread use of a number of these materials in nitric acid recovery systems. The presence of fluorides and chlorides in sufficient amounts to accelerate the attack was suspected. Analyses of the various solutions showed neither of these contaminants to be present in the 200 ppm or higher amounts which are reported to be required to increase the attack by nitric acid on stainless steels.[21]

Hydrogen Chloride and Hydrochloric Acid

In hydrogen chloride and commercial hydrochloric acid exposures, titanium was generally unsatisfactory while zirconium was generally good (Table 9).

In 32 percent acid, titanium was attacked rapidly, but in 36 percent acid the attack was a good deal less rapid. This difference very likely is due more to a difference in the amount of residual chlorine in these by-product acids than to a difference in concentrations. The 32 percent acid normally contains less chlorine than the 200 ppm reported in the 36 percent acid.

In dichlorobenzene containing 4-5 percent anhydrous hydrogen chloride at 355 F, titanium was fairly resistant. In a perchlorethylene-carbon tetrachloride mixture saturated with hydrogen chloride and chlorine at 250 F, it failed rapidly. The amount of water present (20-30 ppm) was below that required to maintain the passivity of titanium in the presence of chlorine.

Zirconium was resistant to the concentrated acids although some attack under the spacer and at the specimen edges was found. The effect of chlorine no doubt accounts for the difference in attack in Exposure 3 against that in Exposures 4 and 5.

In the presence of 4-5 percent dry hydrogen chloride, zirconium exhibited good resistance. No explanation can be offered for the high rate of attack it suffered in Exposure 1, where essentially dry chlorine and hydrogen chloride are present.

The test described in Exposure 5 was an afterthought. It was conducted primarily to provide information on the performance of tantalum in hydrochloric acid containing 200 ppm chlorine. Titanium was included to see if that much chlorine might provide protection in an exposure where titanium otherwise would fail rapidly. A titanium specimen plated with five-millionths of an inch of platinum was also included.

As shown, tantalum was not affected appreciably. Titanium apparently received some protection from the chlorine. The platinum-plated titanium proved to be quite resistant in this short-term exposure. (All the specimens in Exposure 5 have been returned for a long-term test).

Monel and Hastelloy C showed up well in Exposures 1 and 2 under anhydrous conditions, Hastelloy B and Chlorimet 2 were resistant to the hydrogen chloride in Exposure 2.

In 32 percent acid, Hastelloy B and Chlorimet 2 were resistant while Hastelloy C was not. But in the 36 percent acid, both Hastelloy B and Chlorimet 2 were severely attacked. In view of this evidence of the far more aggressive nature of the 36 percent acid, whether due to high concentration or the presence of chlorine, zirconium's performance in this acid is somewhat easier to understand.

Salt Solutions

Salt solutions of several types, and at various temperatures, had little or no effect on titanium or, with one exception, on zirconium (Table 10).

Titanium was extremely resistant to sea water, even at high velocity, to sodium chloride and potassium chloride brines at 140 to 160 F, and to a low pH sodium chloride brine at 165 F (Exposure 7).

Zirconium was almost equally resistant to all but the low pH brine, in which it suffered a high corrosion rate and severe pitting and spacer attack. This low pH brine proved very aggressive toward all the materials tested with the exception of titanium.

Hastelloy C was generally good in the sodium and potassium chloride brines (Exposures 3 to 6). Monel and nickel had low corrosion rates in these same exposures but showed some tendency toward attack under the spacers.

Steel suffered generally high corrosion rates with some pitting tendency evident.

Miscellaneous Exposures

The results of tests in some miscellaneous exposures are shown in Table 11.

Titanium was very resistant to river water containing chlorides, organics and intermittent chlorine, to an alkaline zirconyl hydroxide slurry, and to 5 percent sulfur dioxide at 425 F. It showed good resistance to a 200 F waste gas containing chlorine, moisture, sodium hydroxide and sodium chloride and fairly good resistance to 430 F steam which occasionally contains small amounts of ammonia, sodium hydroxide, sodium chlorate and sodium chloride.

Zirconium was excellent in the river water and the 430 F steam exposures. It showed a moderate corrosion rate in the hot 5 percent sulfur dioxide and was severely attacked in zirconium tetrachloride and in the waste gas containing wet chlorine.

The relative severity of these miscellaneous exposures can best be judged by a study of their effect on the other materials tested.

Atmosphere Exposures

Table 12 shows the corrosion resistance of a number of materials in several types of atmospheres, mostly chlorine-containing. Titanium proved exceptionally resistant in all cases, while zirconium was satisfactory only in the least aggressive of these environments.

The severity of these exposures can be judged by the behavior of steel. In Exposure 1, the contaminant was steam laden with sodium hydroxide. Titanium, zirconium, stainless 316 and the other materials including steel, were all satisfactory. In Exposures 2 and 3, the contaminants were hydrogen chloride and chlorine in moist air, a severe condition as judged by the corrosion rate of steel. Only titanium was resistant. In Exposure 4, moist hydrogen chloride vapors created a severe exposure condition as shown again by the attack on steel and again titanium was the only satisfactory material.

TABLE 10—Exposures in Salt Solutions

Exp. No.	Corrosion Rates, Mils Per Year										
	Ti	Zr	Ni	Monel	Hast. C	316	Al 6061	Cu-Ni 70-30	Cu-Ni 90-10	Aluminum Brass	Mild Steel
1	0.003	0.01	....	4	....	0.03	0.5[5]	5	10	10	84
2[10]	Nil	0.05	....	17[2,5]	....	0.02[2,6]	2[2,5]	9[4,8]	10[5]	_[10]	212[2,7]
3	Nil	0.001	0.07[7]	0.08[6]	Nil	....	....	....	....	.....	215
4	Nil	0.004	0.05[7]	0.6[6]	Nil	....	....	....	....	.....	34
5	0.006	0.007	0.05[6]	0.05[6]	0.002[6]	....	....	....	....	.....	83
6	0.002	0.009	0.1[6]	0.02[6]	0.002	....	....	....	....	.....	11
7	Nil	0.01	0.05[6]	0.04[6]	0.02	....	....	....	....	.....	26
8	0.01	19[5,8]	>97[1]	>90[1]	12 0.1[9]	....	....	....	....	.....	>190[1]

[1] Specimens were completely consumed during the test.
[2] One of two duplicate specimens was lost.
[3] Slight or shallow pitting attack.
[4] Moderate pitting attack.
[5] Severe pitting attack.
[6] Slight attack under spacer.
[7] Moderate attack under spacer.
[8] Severe attack under spacer.
[9] One of two specimens suffered severe pitting and under-spacer attack.
[10] This test will be repeated since some specimens were lost and some were obviously abraided against the pipe wall.
Corrosion rates were calculated on undamaged specimens.

Exposure No.	Environment	Temperature	Duration of Test, Days
1	Sea water saturated with CO_2	75 F	160
2	Sea water, very high velocity	75 F	212
3	Saturated NaCl brine, pH-10.4	135 F	125
4	Saturated NaCl brine	70 F	208
5	Saturated NaCl brine	140 F	169
6	Saturated KCl brine	140 F	183
7	Saturated NaCl brine	160 F	163
8	20% NaCl brine, pH-2.0-4.5	165 F	73

TABLE 11—Miscellaneous Exposures

Exp. No.	Corrosion Rates, Mils Per Year																			
	Ti	Zr	Ni	Monel	Inconel	Nionel	Hast. C	Chlorimet 2	Chlorimet 3	304	316	310	Carpenter 20	430	Duriron	Durichlor	Lead	Al	Copper	Mild Steel	Cast Iron
1	0.002	0.006	0.1	0.1	..	..	..	..	..	Nil	Nil	..	..	..	..	..	..	0.1[3]	1	14	..
2	Nil	..	0.03	0.02	..	..	..	..	..	Nil	0.02	..	..	..	..	..	..	..	..	4	..
3	..	>240[1]	..	..	..	..	..	..	..	..	32[7]	36	..	18	..	..	..	..	..	..	..
4	1	7	14[4]	68	6	1	..	..	..	2	2	..	..	..	0.3	..	..	..	..	14	15
5	2	6[5]	>43[1]	..	..	..	10[6]	>165[1]	..	....	..	..	..	..	..	20[4]	>66[1]	..	..	85	..
6	2	4[3]	>74[1]	..	..	..	37	..	29	....	..	..	..	..	..	>355[2] 37[4]	>115[1]	..	..	..	..
7	..	0.6	2	3	..	..	0.4	..	..	....	0.6	..	0.3	..	..	..	..	..	..	..	..

[1] Specimens were completely consumed during the test.
[2] One of two duplicate specimens was completely consumed.
[3] Slight or shallow pitting attack.
[4] Severe pitting attack.
[5] Specimens were perforated by very severe pitting attack.
[6] Specimens were perforated by very severe attack under spacer.
[7] Specimens were visibly cracked.

Exposure No.	Environment	Temperature	Duration of Test, Days
1	River water containing chlorides, various organics and intermittent chlorine	90 F	169
2	Zirconyl hydroxide slurry containing ammonium sulfate and ammonia	125 F	115
3	Zirconium tetrachloride vapor containing a small amount of hydrogen chloride	100 to 1700 F (batch operation)	25
4	Air containing about 5% of sulfur dioxide, 1% of sulfur trioxide and 6% of moisture	425 F	68
5	Waste gas containing chlorine, moisture and droplets of sodium hydroxide-sodium chloride solution	200 F	167
6	Duplicate test in Exposure No. 6		98
7	Steam containing occasional traces of ammonia, sodium hydroxide, sodium chlorate and sodium chloride	430 F	129

In Exposures 5 to 11, the atmospheres were typical of those in various locations in electrolytic chlorine plants (i.e., moist air containing varying amounts of chlorine). As could be expected, titanium was virtually unaffected in all these exposures. Zirconium showed some very low corrosion rates but tended to pit slightly. The corrosion rates for steel varied considerably, probably with the amount of chlorine in the atmosphere. Nickel, Monel, Hastelloy C, Chlorimet 3 and copper were all very good. Aluminum and the stainless suffered low rates but were inclined to pit steels.

Conclusions

While titanium demonstrated excellent resistance to a wide variety of corrosive environments, it was particularly outstanding in: (1) wet chlorine gas, under all conditions up to 205 F, (2) solutions containing chlorine, (3) hypochlorous acid and hypochlorites, (4) nitric acid from 35 to 65 percent up to 265 F, (5) calcium chloride up to 55 percent and 235 F, (6) atmospheres containing chlorine and hydrochloric acid.

Zirconium also showed excellent resistance to a number of environments among which were: (1) hydrochloric acid up to 36 percent, (2) dry chlorine, (3) sodium hydroxides in all concentrations up to 73 percent at 280 F, (4) nitric acid in all concentrations up to 65 percent and 265 F, (5) calcium chloride solutions up to 55 percent and 235 F.

Titanium equipment, ranging from valves to heating coils to nitric acid reboilers, is being evaluated in all the exposures in which titanium has proved outstanding. Zirconium is being used in dry chlorine and in strong hydrochloric acid service, as valve stems in control valves, and is under consideration for use in other exposures where it showed low corrosion rates. These equipment installations, most of which have been in service six months or more, have thus far confirmed the results of the specimen tests.

This program, like many such investigations, raised a number of questions which remain to be answered. Anodic protection of titanium has received some attention. The effect of chlorine on the corrosion resistance of titanium in acids other than sulfuric acid and especially in hydrochloric acid is being investigated. Additional work with platinum-plated titanium is under way. A number of the original tests are not complete as this is being written. Additional tests have been or will be begun to take care of deficiencies in the original program, especially in regard to omission of comparison specimens of other materials and length of exposure.

Results obtained in this program largely substantiate previously reported data, not only on titanium and zirconium but on most other materials as well. A few exceptions were found, however. The erratic performance of titanium and zirconium in boiling 62 percent and 72 per-

TABLE 12—Atmospheric Exposures

Exp. No.	Corrosion Rates, Mils Per Year										
	Ti	Zr	Ni	Monel	Hast. C	Chlorimet 3	304	316	Al 6061	Copper	Mild Steel
1	0.002	0.03	....	0.01	....	.....	....	0.0002	0.05[1]	0.09	3
2	0.005	6[3,5]	6[4]	13	....	.....	7[7]	13[7]	33[7]	20[5]	38
3	0.01	6[3]	8	26	....	.....	10[3,6]	8[3,6]	96	55[6]	90
4	0.004	4[2]	5[1]	8[1]	....	.....	3[2,6]	4[3,6]	18	21[5]	58
5	0.004	0.02	0.05	0.1	....	.....	....	0.05	0.2[1]	0.2	8
6	Nil	0.04[2]	0.02	0.1	....	.....	0.4[2]	0.3[5]	0.5	2	6
7	Nil	0.3[2]	0.2	0.9	....	.....	2	1[2,5]	6[3]	2	7
8	0.0002	0.009	0.003	0.03	0.001	0.006	0.004	....	0.3	0.09	4
9	0.0003	0.09[2]	0.8	1	0.001	0.2	....	....	6	1	14[1]
10	0.0002	0.1	0.3	0.4	0.01	0.01	0.2[1,4]	....	2	0.8	30
11	0.002	0.003	0.03	0.05	....	.....	....	0.02	0.2[1]	0.6	3

[1] Slight or shallow pitting attack.

[2] Moderate pitting attack.

[3] Severe pitting attack.

[4] Slight attack under spacer.

[5] Moderate attack under spacer.

[6] Severe attack under spacer.

[7] Specimens perforated by severe pitting attack.

Exposure No.	Environment	Temperature	Duration of Test, Days
1	Moist air containing alkaline contaminants	Ambient	166
2	Moist air containing HCl and Cl_2	Ambient	118
3	Moist air containing HCl and Cl_2	Ambient	92
4	Moist air containing HCl	Ambient	137
5	Moist air containing Cl_2	Ambient	166
6	Moist air containing traces of Cl_2	92 F	214
7	Moist air containing traces of Cl_2	98 F	214
8	Moist air containing traces of Cl_2	Ambient	195
9	Moist air containing Cl_2	Ambient	195
10	Moist air containing Cl_2	Ambient	195
11	Moist air containing Cl_2	120 F	166

cent calcium chloride was surprising, particularly in the case of titanium which was reported as being resistant up to 80 percent concentration. Titanium's excellent resistance to sodium hydroxide solutions up to 73 percent was another surprise, mostly because previous data stopped at 40 or 50 percent concentrations. In sulfuric acid containing chlorine, titanium was resistant up to 72 percent concentration against a known useful resistance only up to 5 percent in pure acid. Zirconium, on the other hand, was adversely affected by chlorine in sulfuric acid below 60 percent concentration.

Titanium and zirconium obviously possess corrosion-resisting properties which can and will be exploited by the chemical industry. In some cases, these properties are so outstanding it appears a more general use of these two materials can be justified readily on a purely economic basis.

Acknowledgment

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Any discussion of this article not published above will appear in the December, 1959 issue

Corrosion of Types 316 and 317 Stainless Steel By 75 Percent and 85 Percent Phosphoric Acid*

By ALBERT R. MORGAN, JR.

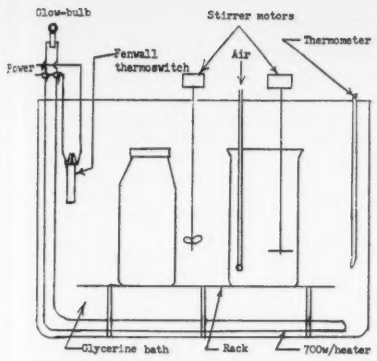

Figure 1—Schematic diagram of laboratory constant temperature bath apparatus.

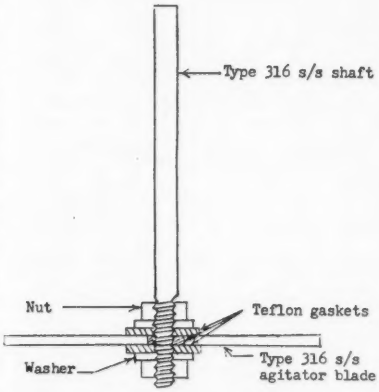

Figure 2—Schematic diagram of agitator blade.

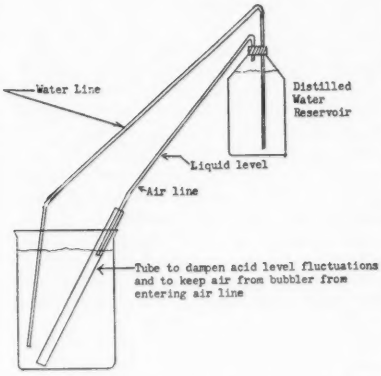
Water Line
Distilled Water Reservoir
Liquid level
Air line
Tube to dampen acid level fluctuations and to keep air from bubbler from entering air line

Figure 3—Schematic diagram of constant level controller for aerated and agitated acid samples.

TABLE 1—Chemical Analysis of Type 316 Stainless Steel Used for Laboratory Test Specimens

Element	Content, Percent	
	Rolled	Cast
Chromium	17.58	18.95
Nickel	11.33	9.65
Molybdenum	2.49	2.75
Carbon	0.08	0.05
Manganese	....	0.60
Silicon	....	0.96

Introduction

THE AUTHOR'S company produces white phosphoric acid in patented wetted wall furnaces made entirely of Types 316 and 316 ELC stainless steel. The acid produced and recirculated in the furnace is usually 75-76 percent H_3PO_4 by weight although 78 percent and 85 percent acids also are produced. As considerable heat is liberated in the combustion of phosphorus, and this heat is absorbed by the acid, it was necessary to determine the limiting acid temperature which could be used in order to protect the equipment.

The corrosion test work described in this paper was done first on a laboratory scale and then under pilot and plant conditions.

Laboratory Test Work

A laboratory constant temperature bath was set up as shown in Figure 1. The basin was cut from a ten gallon borosilicate carboy and filled to the desired level with glycerine, which was heated by a 700 watt stainless steel immersion heater controlled by a Fenwall thermoswitch. An agitator circulated the glycerine and the temperature was measured with a simple mercury thermometer. The Type 316 stainless steel test specimens were placed in jars and beakers of the various acid samples; these jars were about four inches above the bottom of the bath on a stainless steel rack. The specimens were weighed before immersion and at 2, 8, and 16 days from the starting time of each run. The temperature runs studied were at 85, 100, and 115 C. After each temperature run the specimens also were examined under a microscope for signs of corrosion.

The analyses of the rolled and cast Type 316 stainless steel used in these tests are listed in Table 1. The 1 inch x 2 inch x 1/16 inch rolled specimens were all cut from the same sheet stock, and the cast specimens (1½ inch x ½ inch x ¼ inch) were sawed from one casting. A bead of 316 ELC weld metal was laid across the face of several of the rolled pieces; these are referred to as "welded" specimens. All original specimens were cleaned by immersing in aerated 75 percent furnace acid at 70 C for 24 hours before the initial weighing. Specimens were used in successive temperature runs until discolorations or etchings visible to the eye were noted.

One master sample of furnace run 76 percent phosphoric acid was used to prepare all the acid samples. An analysis of this acid is shown in Table 2. For the 85 C run, the 85 percent acid was made by adding P_2O_5 to the 76 percent acid. For the 100 and 115 C work, 76 percent acid was concentrated to 85 percent by heating.

Both concentrations of acid were used under two different conditions: aerated—agitated, and static at each temperature. The static samples were placed in sealed jars, while the aerated and agitated samples were contained in open beakers. Aeration was maintained by passing filtered air through a fritted glass sparger in the bottom of each beaker. The impellers were 1 inch x 2 inch pieces of rolled 316 stainless steel, rotated at about 600 rpm. The impellers were electrically insulated from the shafts by fluorocarbon gaskets to preclude any couple effects

About the Author

ALBERT R. MORGAN JR., has a BS in chemistry from the University of California. Following a World War II tour as an army ordnance captain, and a year of graduate work at University of California in Chemical Engineering, he joined Food Machinery and Chemical Corp. Since 1952, he has been Process Department Superintendent at Westvaco Mineral Products Division, Newark, California and has specialized in magnesia, refractory and phosphorus technology. He is a member of AIChE, ACS and ASTM.

Abstract

The corrosion of Types 316 and 317 stainless steel by 75 percent and 85 percent H_3PO_4 was studied in the 78-115 C range in laboratory, pilot and plant scale tests. Corrosion rates were found to be considerably affected by acid temperatures and by testing conditions. Although laboratory data indicated a maximum permissible temperature of over 115 C for 316 stainless steel in either 75 percent or 85 percent H_3PO_4, and pilot data indicated a maximum permissible temperature in excess of 105 C in 75 percent acid, extensive plant data showed 316 and 316L to be acceptable (< 5 mpy penetration) only at 100 C or less. Type 317 proved more resistant than 316L in both pilot and plant tests. Aerated and agitated samples showed lower corrosion rates than stagnant samples. Welded samples were as resistant as solid sheets in all tests. Cast 316 proved as resistant as rolled 316. 4.3.2

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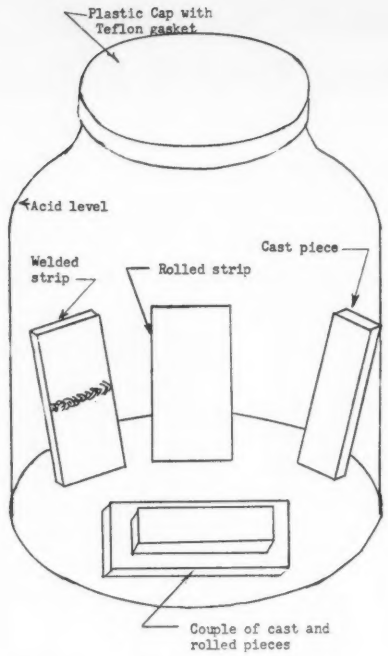

Figure 4—Arrangement of specimens in sample jars and beakers.

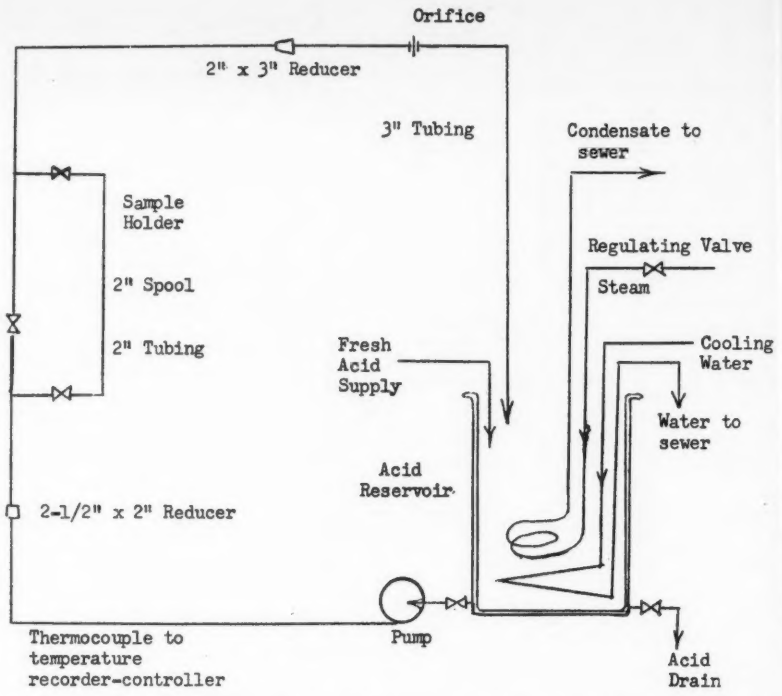

Figure 5—Schematic diagram of pilot test corrosion equipment. All lines had Type 316 stainless steel tubing.

TABLE 2—Analysis of Furnace Run Phosphoric Acid

Item	Content
H_3PO_4	76.26 Percent
SO_3	0.00 Percent
Cl^-	6.2 ppm
F^-	0.3 ppm
Fe	4.0 ppm

(Figure 2). To minimize concentration changes by evaporation from the open containers, the liquid level was maintained constant by the addition of distilled water through siphon devices as illustrated in Figure 3. The concentration was checked occasionally throughout the run and again at the end.

Each of the four acid samples contained five steel specimens. The rolled, cast, and welded specimens were placed on end leaning against the glass container walls. A rolled and a cast specimen were also placed in direct contact on the bottom of the vessels with the ½ inch x 1½ inch side of the cast piece resting on the rolled piece. A diagram of this specimen arrangement is shown in Figure 4. All of these specimens as well as the two impellers in the aerated and agitated samples were weighed before immersion and after 2, 8, and 16 days exposure.

The average corrosion rates as calculated from the weight losses and the areas of the specimens are listed in Tables 3 through 6. The average penetration of the rolled specimen after 384 hours at 85, 100 and 115 C in static 76 percent phosphoric acid was 0.05, 1.4 and 6.7 mils per year, respectively, while the similarly treated cast specimen averaged 0.05, 1.1, and 4.9 mpy. The attack by the 76 percent aerated and agitated acid was considerably less, averaging 0.00, 0.4 and 0.7 mpy for the rolled specimen and 0.01, 0.07, and 0.7 mpy for the cast piece at the 85, 100 and 115 C temperature levels, respectively. Penetrations by 85 percent furnace acid were similar to those with 75 percent furnace acid, indicating little effect due to acid concentration in this range.

The corrosion rates of the cast and rolled Type 316 stainless steels were roughly similar for any given set of conditions in a given acid. The welded pieces showed penetration rates similar to those of the rolled specimens. In the aerated and agitated acids the Type 316 agitator blades exhibited no more penetration than the corresponding stationary rolled piece. In short, no accelerated corrosion rates were exhibited by couples, welds, or velocity in the aerated-agitated tests. The stagnant samples appeared

TABLE 3—Corrosion of Type 316 Stainless Steels in Static 75 Percent H_3PO_4

Temp, Degrees C	Time, Hours	Average Penetration, mpy				
					Couple	
		Rolled	Cast	Welded	Cast	Rolled
85	50	0.00	0.00	0.00	1.81	0.21
	194	0.04	0.03	0.02	0.99	0.00
	386	0.05	0.05	0.07	0.11	0.07
100	50	0.64	0.11	0.39	0.64	0.15
	193	0.93	0.59	0.66	1.01	0.83
	384	1.42	1.12	1.17	1.32	3.5
115	48	3.3	0.55	3.5	15.3	6.1
	192	5.4	7.8	4.5	8.3	4.7
	383	6.7	4.9	6.1	8.5	7.0

TABLE 4—Corrosion of Type 316 Stainless Steels in Agitated and Aerated 75 Percent H_3PO_4

Temp, Degrees C	Time, Hours	Average Penetration, mpy					
					Couple		
		Rolled	Cast	Welded	Cast	Rolled	Agitator
85	50	0.58	0.85	1.55	1.33	0.97	1.09
	194	0.00	0.00	0.23	0.23	0.22	0.25
	386	0.00	0.01	0.09	0.10	0.08	0.11
100	50	....	0.48	....	0.53	0.27	0.22
	193	0.39	0.65	0.26	0.43	0.35	0.38
	384	0.44	0.69	0.41	0.58	0.46	0.50
115	48	2.4	1.99	2.9	1.16	1.96	1.87
	192	0.83	0.64	0.83	0.35	0.63	0.73
	383	0.71	0.65	0.77	0.47	0.58	0.65

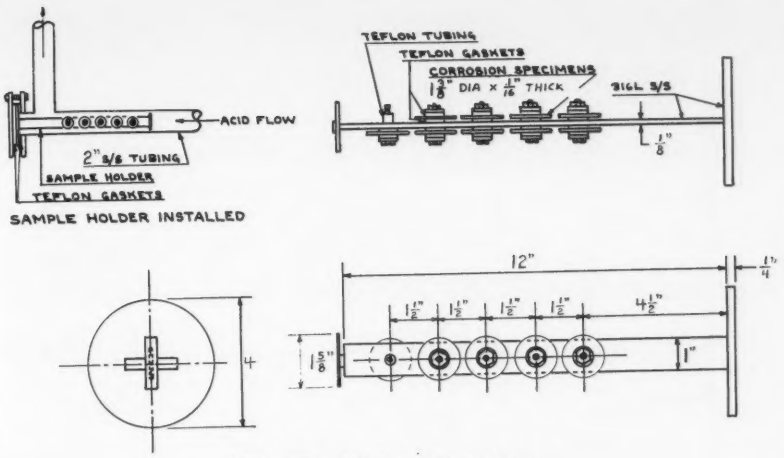

Figure 6—Sample holder used in pilot plant.

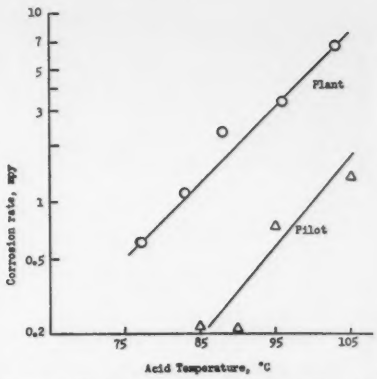

Figure 7—Plant and pilot data for Type 316L stainless steel versus 75 percent phosphoric acid.

definitely to corrode at a more rapid rate, however. Microscopic inspections of the specimens after each temperature run showed no evidence of localized pitting or accelerated corrosion. Some of the metals in the static samples showed a surface discoloration, and, at the higher temperatures, a minor corrosion of the cast pieces was noted at stressed points (i.e., on sawed edges.)

The above laboratory experimental program was repeated with several other grades of 75 percent and 85 percent acids including commercial and food grade. The results under aerated and agitated conditions were essentially the same as those reported here. A block at chloride levels of 25-75 ppm as NaCl also showed no change in corrosion rate (in mpy) from this work, on aerated and agitated samples. However static rolled samples at the higher chloride levels corroded at a 15-20 mpy rate at 115 C in a 400 hour test and in addition pitted. Static cast samples under same conditions corroded only 2-4 mpy with no pitting.

Pilot Test Work

A pilot test was then set up to simulate as closely as possible the conditions of the production furnaces. The equipment consisted of a tank, circulating pump and lines, temperature regulating and controlling equipment, and an orifice. All equipment in contact with the acid was 316 stainless steel. The layout of the test equipment is shown in Figure 5. The sample holder used is shown in Figure 6. It was constructed of 316 stainless steel and fluorocarbon tubing and gaskets were used to insulate the specimens. The exposed area of a specimen was about 2.5 sq. inches.

An acid velocity of 895 fpm was picked for the initial studies. The discharge of the circulating line was above the surface of the acid in the reservoir so as to obtain some aeration. In order to minimize the influence of any accumulative corrosion products, the 75 percent plant acid used was changed daily.

The samples were first conditioned in the acid for approximately five days prior to the test. The samples were then cleaned with a detergent, rinsed, dried with acetone, and weighed before the test started. The test period at each temperature was approximately 30 days and every run was made in duplicate, with duplicate samples on each run. At the end of this time, the samples were again cleaned and weighed. The composition of the stainless steel coupons is shown in Table 7. Both welded and non-welded specimens of Type 316 ELC stainless steel were studied at temperatures of 78, 85, 90, 95, and 105 C. Acid velocities of 895-1015 fpm were used at 78 C and 90 C.

During the test period, the pump impeller, lines, orifice, tank and steam coils were inspected and no particular corrosion was noted on the equipment.

The results of these pilot tests are summarized in Table 8 and Figures 7 and 8. Average penetration rates by 75 percent H_3PO_4 on Type 316 ELC stainless steel in the pilot test apparatus at 895 fpm were 0.2 to 1.3 mpy for the 78 C and 105 C temperature range. Welded coupons showed about the same corrosion rates at the higher temperatures; 0.2 to 1.8 mpy. Type 317 showed lower corrosion rates than Type 316 by factor of approximately one-half.

Plant Test Work

Plant test work then followed and consisted of suspending 316, 316 ELC and 317 stainless steel test coupons (Table 9) at various points in a plant operating furnace and noting the corrosion rate (weight loss) over the exposure time for the various acid temperatures checked (77 C-103 C), as shown in Table 10. All determinations in each run were made in duplicate, and, in addition, the entire 96 C and 103 C runs were made in duplicate. In all cases the results checked very well. The sample holder used in the plant tests is shown in Figure 9. These holders were constructed of 316 ELC stainless steel and fluorocarbon tubing was used as spacers and insulators. The exposed area of the samples was about 7 to 10 square inch. Exposure times of from 382 to 587 hours were used in each run and the same samples were used for consecutively higher temperature runs. An initial conditioning period of 273 hours was used on the lowest temperature run. The corrosion rates of Type 316 ELC stainless steel were 2.3

TABLE 5—Corrosion of Type 316 Stainless Steels in Static 85 Percent H_3PO_4

Temp, Degrees C	Time, Hours	Average Penetration, mpy				
		Rolled	Cast	Welded	Couple Cast	Couple Rolled
85	50	0.61	0.37	0.67	1.22	0.24
	194	0.31	0.26	0.29	0.69	0.42
	386	0.14	0.14	0.16	0.23	0.34
100	50	...	...	0.03	...	0.18
	193	1.58	1.41	1.31	2.1	2.4
	384	1.19	0.94	1.05	2.5	3.3
115	48	2.7	1.99	3.0	9.1	2.7
	192	4.0	1.95	4.8	4.3	2.8
	383	5.7	1.40	5.3	5.0	4.1

TABLE 6—Corrosion of Type 316 Stainless Steels in Aerated and Agitated 85 Percent H_3PO_4

Temp, Degrees C	Time, Hours	Average Penetration, mpy					
		Rolled	Cast	Welded	Couple Cast	Couple Rolled	Agitator
85	50	1.12	1.17	0.23	0.69	0.18	0.16
	194	0.31	0.27	0.09	0.19	0.09	0.07
	386	0.17	0.11	0.02	0.10	0.00	0.00
100	50	....	....	....	....	....	....
	193	0.26	0.50	0.31	0.44	0.26	0.26
	384	0.83	0.60	0.38	0.62	0.40	0.38
115	48	1.87	1.11	1.14	2.1	1.61	1.73
	192	0.82	0.83	0.72	0.97	0.88	0.78
	383	0.67	0.75	0.58	0.69	0.66	0.66

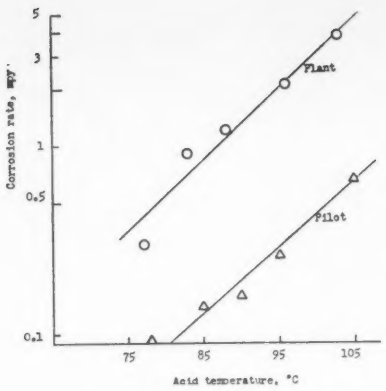

Figure 8—Plant and pilot data for Type 317 stainless steel versus 75 percent phosphoric acid.

TABLE 7—Composition of Stainless Steels Used in Pilot Corrosion Study

Element	Content in Steel Percent	
	316 ELC	317
Carbon	0.02	0.06
Chromium	17.97	19.64
Nickel	12.87	14.30
Molybdenum	2.68	3.50

mpy at 88 C, 3.4 mpy at 96 C, and about 6.6 mpy at 103 C. Cast 316 stainless steel showed lower average penetrations (4.4 mpy average penetrations at 103 C) than the Type 316 ELC. This tendency also was observed in the laboratory tests. Type 316 corroded at a rate of approximately 7.2 mpy at 103 C. The average penetrations in the plant environment were about ten times as severe as the laboratory tests and about five times as severe as the pilot tests at the same temperature. Laboratory tests had indicated that at 120 C, aerated and agitated 75 percent H_3PO_4 might be handled satisfactorily; plant tests, however, indicated an upper limit of about 100 C. Reasons for the divergent data are probably due to different testing conditions, possibly to small differences in the formulation of the alloys tested and to small composition differences in the acids.

Based on these results, 100 C has been established as the maximum usable temperature in plant 316 and 316 ELC process equipment, pending results of further tests at higher temperatures now in progress.

Type 317, as in the pilot tests, showed greater resistance than 316 and 316L with corrosion rates of about 4 mpy compared to 6-7 mpy.

This discussion covers work done to date. Further plant scale work in the 105-115 C region is planned for the near future.

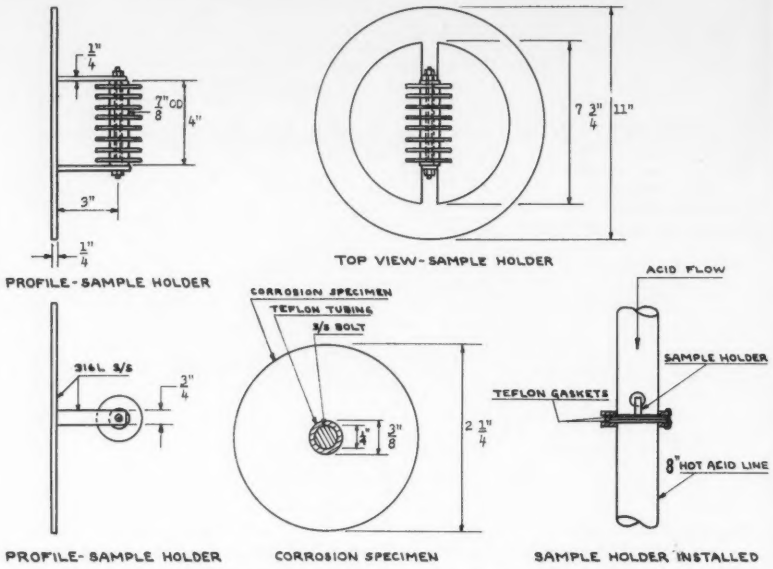

Figure 9—Sample holder used in furnace hot acid line.

TABLE 8—Corrosion of Various Alloys in 75 Percent H_3PO_4
(Pilot Data)

Metal	Corrosion Rate in mpy at Indicated Temperatures*				
	78C	85C	90C	95C	105C
316 ELC	0.28	0.22	0.21	0.64	1.31
316 ELC (Welded)	0.20	0.58	0.34	0.77	1.76
317	0.09	0.14	0.16	0.26	0.65
Carpenter 20	0.18	0.25	0.22	0.58	0.90
Hastelloy F	0.38	0.52	0.39	0.87	1.33
Hastelloy C	0.89	1.23	1.34	2.18	4.60
Alloy 803	0.19	0.26	0.30	0.54	1.26

* Acid velocity 895 fpm, 726-793 hrs exposure at each temperature.

TABLE 9—Analyses of Stainless Steel Specimen
(Plant Tests)

Type of Steel	Constituent, Percent				
	C	Mn	Ni	Cr	Mo
316L 316L Welded	0.04	1.9	13.3	17.3	2.1
316 Cast	0.07	0.9	7.8	19.0	2.2
316 316 Welded	0.05	1.9	13.5	17.4	2.0
317 317 Welded	0.05 0.04	1.6 1.6	14.2 13.8	17.6 18.5	3.2 3.3

TABLE 10—Corrosion of 316 and 317 Stainless Steel in 75 Percent H_3PO_4
(Plant Data)

Type of Steel	Corrosion in mpy at Indicated Temperatures*				
	77C	83C	88C	96C	103C
316 ELC	0.6	1.1	2.3	3.4	6.6
316 ELC Welded	0.6	1.3	2.4	3.5	6.7
316	..	..	1.4	3.5	7.2
316 Welded	..	..	1.4	3.9	8.0
316 Cast	..	..	..	2.1	4.4
317	0.3	0.9	1.2	2.1	3.7
317 Welded	..	1.0	1.3	2.5	4.0

* Exposure times of 365-587 hours on 83C-103C samples; 683 hours on 77C run.

NATIONAL ASSOCIATION of CORROSION ENGINEERS

Summary of Replies to Questionnaire On Handling of Chlorine Mixtures

NACE TECHNICAL COMMITTEE REPORT
Publication 59–15

A Report of NACE Task Group T-5A-4 on Chlorine*

Abstract

A summary is made of replies to questionnaires on handling of various chlorine mixtures. Information reported practices and findings of nine companies on the following matters: materials used for handling cell gases (with particular reference to thermosetting resins reinforced with asbestos and/or graphite and chemically resistant ceramic-ware); materials used for handling sniff gas (with particular reference to steel); materials used for handling other chlorine mixtures; suitability of steel lines for liquid and gaseous chlorine in terms of velocity; materials used for handling liquid and gaseous chlorine in valves, pumps and gaskets; experience with PVC in various chlorine environments. 4.3.5

Introduction

THIS REPORT constitutes a summary of replies to a questionnaire on handling various chlorine mixtures sent to members of Task Group T-5A-4 on Corrosion in the Chlorine Industry and to other interested parties. Replies were received from nine companies with several replying with information from more than one plant, making a total of twelve replies received. This information is summarized in Table 1. The companies replying are as follows:

Columbia-Southern Chemical Company—plants at Corpus Christi, Texas, and Barberton, Ohio

Diamond Alkali Company

Frontier Chemical Company

Hooker Electrochemical Company

Pennsalt Chemical Company — plants at Tacoma, Washington; Portland, Oregon; Calvert City, Kentucky

Stauffer Chemical Company

Union Carbide Chemicals Company — Texas City, Texas

Westvaco Chlor-Alkali Company

Wyandotte Chemicals Corporation

The questionnaire stemmed from desire on the part of the committee to correlate the experiences of both manufacturers and users with regard to materials of construction for handling various chlorine mixtures. The earlier activity of the committee in collecting information leading to the bibliography on "Corrosion from Chlorine" served as background for this work.

Chlorine Environments in Which Materials Used

As noted, the questionnaire contains six particular topics:

1. Materials for handling cell gases (with particular reference to thermosetting resins reinforced with asbestos and/or graphite and chemically resistant ceramic-ware.)
2. Materials for handling sniff gas (with particular reference to steel.)
3. Materials for handling any other chlorine mixtures.
4. Suitability of steel lines for liquid and gaseous chlorine, taking into account velocity effects.
5. Materials for handling liquid and gaseous chlorine in valves, pumps and gaskets.
6. Experiences with PVC in particular chlorine environments.

The fairly divergent results are indicative of several variables at work in the chlorine industry, including such matters as variation in cell type and production rate, operating temperatures, age of the plant, and basic company policy toward materials that would be in the experimental category.

Cell Gases

Thermosetting resins reinforced with asbestos and/or graphite and chemically resistant ceramic-ware were the two materials with which most companies had widest experience in handling cell gases (the gases direct from the electrolytic cells producing the chlorine). Several companies reported that chemically resistant ceramic-ware was unsuitable because of thermal shock failure. The use of epoxy resin and fiberglass to repair the chemically resistant ceramic-ware was noted by two companies. Other satisfactory materials, in addition to modified phenolic resin with asbestos were chemical glass, rubber lined steel, coal-tar impregnated cement-asbestos, PVC and modified PVC at lower temperatures, and steel for dry gas.

Sniff Gas

Sniff gas is the gas removed from tank cars or storage tanks and is generally considered to be high purity chlorine with only small amounts of dry air present. In most all cases, sniff gas is dry and at ambient temperature, free of any organic chlorides. The replies to the questionnaire indicate that most companies have had satisfactory service with steel, while several companies have

* L. W. Gleekman, Wyandotte Chemicals Corp., Wyandotte, Michigan, chairman.

TECHNICAL PAPERS ON CORROSION WELCOMED

Authors of technical papers on corrosion are invited to submit them for review without invitation to the Editor of Corrosion. Write for "Guide for the Preparation and Presentation of Papers" sent free on request to prospective authors.

TABLE 1—Summary of Company Replies to Questionnaire on Handling of Chlorine Mixtures*

Reply Designation	MATERIALS FOR HANDLING CELL GASES. Thermosetting Resins Reinforced With Asbestos and/or Graphite	MATERIALS FOR HANDLING CELL GASES. Chemically Resistant Ceramic-Ware	Materials For Handling Sniff Gas (Steel Pipe)	Materials For Handling Other Cl_2 Mixtures	Suitability of Steel Transfer Lines for Liquid and Vapor Cl_2 in Terms of Velocity	Materials for Valves, Pumps And Gaskets In Handling Liquid And Vapor Cl_2	Experience With PVC In Various Chlorine Environments
"A"	Modified phenolic resin reinforced with asbestos and/or graphite standard.	Suitable, but easily broken due to thermal shock.	Steel suitable.	Chemical glass—suitable for cell gas temperature and below. Glass-lined steel—satisfactory on wet gas at ambient temperature. Thermosetting resins reinforced with asbestos and/or graphite—for cooler shells and tube sheets.	X-heavy seamless steel pipe used; no high velocity problems encountered.	Pumps—Hastelloy C on glands and shaft—steel and cast iron elsewhere. Valves—cast steel body, Hastelloy C stem, 316 trim. Gaskets—rubberized asbestos or fluorocarbon.	Very limited: indicated suitability at lower temperature; appears to suffer initial chlorination and then does not deteriorate further for long periods of time.
"B"	Modified phenolic resin reinforced with asbestos and/or graphite and phenol-formaldehyde resin with asbestos fiber satisfactory.	Satisfactory, but cracks due to thermal shock.	Schedule 80 when gas is dry.	Modified phenolic resin reinforced with asbestos and/or graphite and phenol-formaldehyde resin with asbestos fiber satisfactory. Chemically resistant ceramic-ware satisfactory, but cracks due to thermal shock.	Schedule 80 used, with no velocity problems in usual pipe sizes.	Pumps—none in service. Valves—forged steel body with Hastelloy C trim. Gaskets—rubberized asbestos, rubber-covered lead, lead or fluorocarbon envelope.	No experience.
"C"	Modified phenolic resin with asbestos fiber very satisfactory.	Unsatisfactory due to thermal shock failure; Chemical glass very satisfactory.	Schedule 80 steel for dry gas; Monel trim valves; copper tubing for short connections.	Glass-lined steel—satisfactory on wet Cl_2 at 175 F; rubber lined steel—satisfactory for wet under 100 F no organics; Schedule 80 steel—satisfactory for dry Cl_2 at any concentration.	Schedule 80 used, flanged or welded; pipe sized to give no velocity problems.	Pumps—liquid chlorine pumps satisfactory—steel, carbon and Monel construction. Valve—forged steel body, Monel stems, seats and discs. Gaskets—asbestos only; fluorocarbon satisfactory for rigid joint, satisfactory for cold gas but not for hot wet gas.	Satisfactory under 150 F, 2 years satisfactory service in cool wet Cl_2; requires separate support.
"D"	Phenol-formaldehyde resin with asbestos fiber—7-10 year life with some resin attrition; modified phenolic resin reinforced with asbestos and/or graphite now being used.	Good experience with ceramic tile but suffers thermal shock cracking. Rubber-lined steel—7-10 year life; difficult to patch.	Satisfactory service with mild steel on dry sniff gas.	After H_2SO_4 drying—wrought iron satisfactory after 6 years; steel satisfactory after 5 years.	Schedule 80 used exclusively up to 15M lbs/hr in 1½" pipe—velocity no problem.	Pumps—chlorine pumps satisfactory with 1½ years between overhauls. Valves—mild steel, heavy duty construction; bolted bonnets with deep stuffing boxes. Gaskets—blue African asbestos rubberized.	Satisfactory service in scrubbing and fume system.
"E"	Modified phenolic resin reinforced with asbestos and/or graphite satisfactory.	Fragile and cracks; chemical glass—good.	Satisfactory service with steel.	Chemically resistant ceramic-ware for Cl_2 with weak H_2SO_4; steel for Cl_2 with strong H_2SO_4; modified phenolic resin reinforced with asbestos and/or graphite for Cl_2-H_2O mixtures; polyester for Cl_2-NaCl mixtures if weak Cl_2.	No problem.	Valves—cast steel gates and globes with Monel trim.	No experience.
"F"	Not used.	Satisfactory, but occasional cracking. Chemical glass satisfactory, if no bending stresses; PVC satisfactory for 1 year at 70 F. Coal tar impregnated cement-asbestos satisfactory as cell gas headers for 2 years.	Schedule 80 fair on dry gas; PVC good on dry cool gas; vinylidene chloride resin excellent after 7 years in NaOH absorption; cement-asbestos satisfactory for vent fan exhaust.	Cast iron valve bodies for dry Cl_2 gases; Schedule 80 steel for dry Cl_2 gas 100 F; Monel and Type 316 satisfactory for valve seats and stems on dry Cl_2; thin wall chemically resistant ceramic-ware tubing handling 190 F wet Cl_2 with 56 F H_2O outside Cl_2 at 80 F.	Schedule 80 satisfactory if sized properly; no velocity problem.	Pumps—2-stage vertical pumps for liquid Cl for 5 years. Valves—Cast iron globe with Monel trim for dry Cl_2; gaskets—asbestos sheet.	Satisfactory on dry sniff gas for 5 years; satisfactory on 20° Be' HCl for 4 years 70 F; 2 years life on wet Cl_2 valve at 75 F; satisfactory service on tank linings, coil supports and packages for NaOCl (15% available Cl_2).
"G"		Excellent but cracking from thermal effects. Polyethylene—high mol wt.—stress cracked in 149 days.	Schedule 40 and 80 satisfactory.	Glass satisfactory for wet Cl_2 at 70-80 C; rubber-lined coolers satisfactory for wet Cl_2 at 70-80 C; Schedule 80 steel satisfactory for dry Cl_2 at ambient; chemically resistant ceramic-ware satisfactory for wet Cl_2 at ambient temperature.	Schedule 80 steel seamless satisfactory; no velocity problems.	Pumps—cast steel vertical pump satisfactory on liq. with some steel shaft problems. Valves—cast steel globes with Monel trim for liq. or Cl_2 with 1000 H_2O; all steel satisfactory for Cl_2 with 100 PPM H_2O; Monel check valves for unloading tank cars; fluorocarbon packing in all cases. Gaskets—rubberized asbestos.	None.
"H"		Satisfactory if protected from mechanical shock.	Schedule 40 steel.	Schedule 40 steel excellent after acid drying; bronze flexible hose where required.	No problems.	Pumps—no experience. Valves—cast steel lift plug valve; gaskets—asbestos.	Unplasticized PVC satisfactory for 18 months in wet or dry Cl_2 at 30-150 F.

*Note: Alphabetical order of company designations in table is not necessarily the same as order of company listings in text.

TABLE 1—Summary of Company Replies to Questionnaire on Handling of Chlorine Mixtures*

TABLE 1—Summary of Company Replies to Questionnaire on Handling of Chlorine Mixtures (Continued)*

Reply Designation	MATERIALS FOR HANDLING CELL GASES. Thermosetting Resins Reinforced With Asbestos and/or Graphite	Chemically Resistant Ceramic-Ware	Materials For Handling Sniff Gas (Steel Pipe)	Materials For Handling Other Cl_2 Mixtures	Suitability of Steel Transfer Lines for Liquid and Vapor Cl_2 in Terms of Velocity	Materials for Valves, Pumps And Gaskets in Handling Liquid And Vapor Cl_2	Experience With PVC In Various Chlorine Environments
"I"	No experience.		No experience.	Steel for vaporization and transfer line from unloading area to operating units.	No problem in vapor transfer.	Valves—cast steel valve with Monel trim and Hastelloy C stem. Gaskets—fluorocarbon.	All grades PVC severely attacked at ambient temperature in both liquid and vapor.
"J"	Modified phenolic resin reinforced with asbestos and/or graphite excellent in 4½ years.	PVC and modified PVC satisfactory if supported; schedule 80 steel for dry gas.	Schedule 80 very good.	Hard rubber lined steel for Cl_2 air at 100 F for 4-6 years—organics severely reduced life; glass satisfactory for 4½ years at 150-170 F; cement-asbestos satisfactory for wet Cl_2—air at 80-150 F and 10 PSIG except for joint problems.	No velocity problem with schedule 80 seamless welded or flanged pipe. Most corrosion on exterior due to moisture and frost.	Pumps—vertical pumps satisfactory for liq. Cl_2, slight bearing problem; construction is steel, Hastelloy C and Monel valves—forged steel globe and angle with Hastelloy C trim and Monel stem—also cast steel globe with Hastelloy C and Monel trim. Gaskets—compressed asbestos.	PVC on exit brine from cells at 170-175 F and 0-50 PSIG saturated with Cl_2 is satisfactory but becomes hard and brittle and takes little shock; usually supported well and provided with an expansion joint or flexible coupling. Life expectancy is 3-5 years.
"K"	Modified phenolic resin reinforced with asbestos and/or graphite or phenol-formaldehyde resin with asbestos fiber very satisfactory.	Used extensively—patched by epoxy fiberglass when cracked.	Very good—schedule to match pressure handled.	Phenol-formaldehyde resin with asbestos fiber for wet Cl_2; furfural alcohol resin with asbestos fiber for equipment in chlorination of organic solvent; Hastelloy C for dip tubes in chlorination of caustic; PVC pipe for dip tube in chlorination of NaOH 150 F.	No problem from velocity.	Pumps—no experience. Valves—Cast iron and steel and gas and liquid Cl_2, Monel trim for liquid. Gaskets—asbestos or fluorocarbon.	Used successfully for chlorine water, Cl_2-saturated H_2SO_4 and wet Cl_2 if temperature 150 F and pressures low. PVC valves not used.
"L"	Modified phenolic resin reinforced with asbestos and/or graphite quite suitable in 4 years.	Cracks from thermal shock repaired by mummifying; hard rubber attacked from organics present that condense on surface in three years.	Schedule 80 exclusively on dry gas.	Phenol-formaldehyde resin with asbestos fiber for wet Cl_2 and weak H_2SO_4; Hastelloy C for wet Cl_2 and strong H_2SO_4; vinylidene chloride resin-lined satisfactory if temperature 160 F; chemically resistant ceramic-ware satisfactory if properly supported; rubber-lined as in No. 1 for temperature and organics.	Schedule 80 seamless welded or flanged satisfactory—no velocity problems if sized properly.	Pumps—no experience. Valves—cast or forged steel globe with Hastelloy C trim. Gaskets—rubberized asbestos.	Satisfactory for 3 years in Cl_2 drying system, for cool gas, satisfactory up to 170 F in waste brine saturated with Cl_2 if supported continuously and expansion joints are provided.

* **Note:** Alphabetical order of company designations in table is not necessarily the same as order of company listings in text.

worked with organics such as PVC or vinylidene chloride resin. The thickness of the steel pipe used depends upon the operating pressure and an apparent small corrosion allowance since many companies used Schedule 80 pipe.

Other Chlorine Gas Mixtures

For handling other chlorine gas mixtures such as wet chlorine at lower temperatures, a wide variety of materials are indicated as being used. In addition, for handling chlorine gas in the drying operation where there is sulfuric acid present, use is made of materials such as chemically resistant ceramic-ware, chemical glass, Hastelloy C, and thermosetting resins reinforced with asbestos and/or graphite, depending upon the relative concentration of the moisture and the sulfuric acid. Major considerations here are the presence of organics which may reduce the life of rubber, and the presence of moisture which can have an effect on steel in the presence of sulfuric acid.

Velocity Problems

No problems were indicated due to velocity in handling liquid and gaseous chlorine in steel lines. Most companies use extra-heavy or Schedule 80 for this service, particularly if the sizing was proper. Some corrosion is indicated on the exterior of these lines.

Valve and Pump Materials

Quite a wide variety of materials were used for pumps, valves and gaskets with a general tendency towards high-alloy materials such as Hastelloy C or Monel, where the liquid or gaseous chlorine could be diluted with moisture at stems or packing glands. Steel is widely used as a material of construction. Basically the same tendency holds true on valves, with many valves being made either of cast or forged steel (depending upon pressure) with Hastelloy C or Type 316 stainless trim, depending upon exposure to moisture. Most companies use either rubberized asbestos or fluorocarbon gaskets.

Use of PVC

Only limited experience is indicated with PVC in various chlorine environments; service up to two to four years is indicated under certain specified conditions. Satisfactory service has been achieved in sodium hypochlorite solutions. Normal temperature restrictions for PVC are indicated in that this material must be supported continuously at moderate temperatures and expansion joints provided. Some experiences have been reported where PVC is severely attacked even at ambient temperature in both liquid and gaseous chlorine.

New Materials

Several companies have indicated that work is currently underway on certain new materials that may have promise economically in handling the various gases and liquids. These materials include titanium, polyester reinforced with glass mat, epoxy resin reinforced with glass cloth, and chlorinated polyether resins.

Corrosion in Sour Water Strippers*

By J. F. MASON, JR., and C. M. SCHILLMOLLER

Abstract

This paper discusses the several methods used for stripping foul waters, disposal of waste gas and water, types of corrosion experienced and steps adopted to mitigate corrosion by inhibition, non-metallic coatings and alloy selection. Corrosion test results obtained in operating foul-water treating units also are presented. 4.6.13

Introduction

BECAUSE OF widespread refinery interest in effective means of disposing of sulfide-bearing waters without causing an air or water pollution problem, corrosion control on equipment used to dispose of this corrosive water has become an important problem to avoid high maintenance costs and excessive downtime. Steps adopted to control the several types of corrosion will be discussed.

Processes

The stripping method is the most generally applied process, and is effective in releasing about 80 to 95 percent of the sulfides contained in foul water. In some areas such as the Los Angeles Basin where specifications call for a maximum sulfide concentration in the effluent water of 1 ppm, further chemical treatment is required. One treatment is neutralizing the sour water feed to the tower with sulfuric acid to decompose the sodium, potassium and other stable sulfides, thereby releasing hydrogen sulfide and forming equivalent water soluble sulfates. Neutralization also can be accomplished by addition of hydrochloric acid.

Two other processes, not in the scope of this article, should be mentioned briefly: the air-oxidation process[1] developed by the Shell Oil Company in which sulfides are converted to thiosulfates and disposed of in the sewer; and the biological treatment of phenol containing water[2] to supplement sulfide stripping in areas where regulations do not permit their disposal. For further information the reader is referred to the attached bibliography.[3, 4, 5, 6]

Several stripping media can be used to distill sulfides from the foul water. They are steam, cat cracker waste flue gas, fuel gas including natural gas and combinations of two stripping media. Ammonium sulfides and ammonium-hydrosulfides are the chief water-soluble sulfides present. When heat is added in the tower bottom, these compounds readily decompose, and the pH increases. Significant corrosion problems will occur in the top of the tower and overhead condenser where the highest concentrations of sulfides are present. Consequently, the type of stripping medium employed has relatively little or no bearing on the problems that will be described, or on the solutions discussed here.

Flow Description

A typical flow diagram of the sour water stripping process is shown in Figure 1. The contaminated water streams are collected in a surge drum to prevent hydrocarbons being carried into the stripper because inclusion in the overhead stream could cause difficulties in the combustion furnace or in the sulfur recovery unit. Sour water pumped from the surge drum is preheated to about 160 F by exchange with the hot stripper bottoms and is then introduced at the stripper column top. Natural pH of the incoming feed may be about neutral or as high as 8 to 9, depending on the source of the waters. If necessary, fresh sulfuric or spent alkylation acid is injected to neutralize the feed and spring the stable sulfide compounds.

The stripper column can be 2 to 6 feet in diameter and about 30 feet high and equipped with 5 to 20 bubblecap trays. Turbogrids or an equivalent height of packing with Raschig rings are also used successfully. A low pressure stripping steam is fed into the bottom of the tower. Average operating conditions are as shown in Figure 1: tower pressure 5 to 10 psig, temperatures from 225 to 230 F. Most of the steam is condensed internally by the incoming feed, the bal-

Schillmoller

Mason

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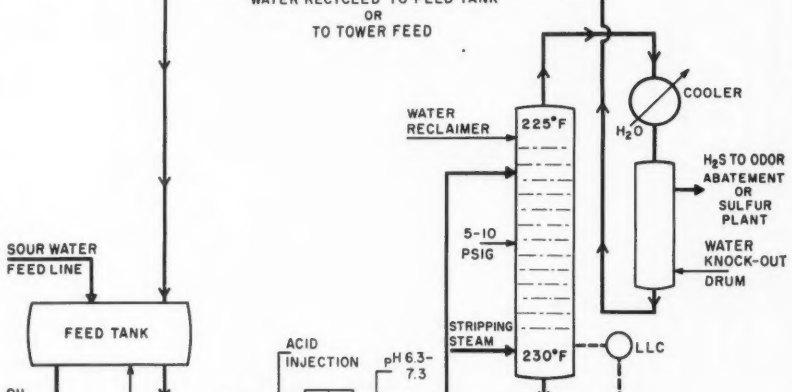

Figure 1—Typical flow diagram of sour water stripping process.

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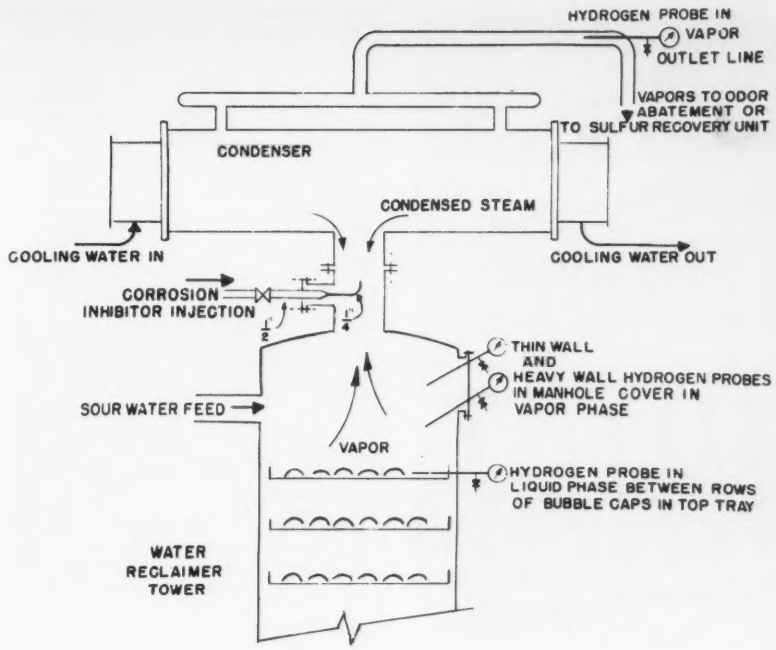
HYDROGEN PROBE IN VAPOR OUTLET LINE
VAPORS TO ODOR ABATEMENT OR TO SULFUR RECOVERY UNIT
CONDENSER
CONDENSED STEAM
COOLING WATER IN
COOLING WATER OUT
CORROSION INHIBITOR INJECTION
THIN WALL AND HEAVY WALL HYDROGEN PROBES IN MANHOLE COVER IN VAPOR PHASE
SOUR WATER FEED
VAPOR
HYDROGEN PROBE IN LIQUID PHASE BETWEEN ROWS OF BUBBLE CAPS IN TOP TRAY
WATER RECLAIMER TOWER

Figure 2—Sketch of water reclaimer tower. Method of inhibitor injection and locations of hydrogen probes are shown.

ance can be condensed by a relatively small condenser.

Figure 2 shows a detailed sketch of a water reclaimer tower illustrating a method of inhibitor injection and locations of hydrogen probes.

Disposal of Water

Several methods can be used for disposal of the stripped waters. In many areas of the country they can be disposed of directly in the sewer; in other parts further treatment is required to take care of objectionable components such as introduction of bacteria to consume phenols.

Several companies have re-used this water in their process as wash waters. Some dilution of corrodent concentrations is effected, but the danger is introduced of raising the concentration of certain corrosive agents in the wash water to 3 or 4 times their natural level. This lowers water washing efficiency and may introduce new problems. As such, it is not advocated.

Disposal of Waste Gas

The sulfide stripping method converts the water pollution problem into an air pollution one. The sulfur bearing gases are usually fed to an odor abatement furnace to be burned with hydrocarbons and converted to sulfur dioxide. Sufficient dilution is generally obtained so that no air pollution or furnace tube corrosion problem results. In areas where this practice is not condoned, hydrogen sulfide gases can be fed to a sulfur recovery unit, directly or via DEA absorbers. Ammonium sulfides are being acidized by one refinery and converted to ammonium sulfates for use in fertilizing solutions.

Corrosion Problem

Most sour water strippers are constructed of carbon steel. Severe corrosion, if it occurs, is limited generally to the tower's top section, overhead transfer line and condenser. In these locations high concentrations of sulfur compounds occur in the liquid and vapor phases. Type and severity of corrosion depend on the pH of the sour water feed, source of water and severity of stripping.

The waters are drawn chiefly from the straight-run, thermal and catalytic cracking units. The latter usually contain considerable amounts of ammonia. At times, waste from the de-salter unit is also included. Besides ammonia and sulfides, a large variety of acids may be stripped, such as formic and acetic acids, cyanides, carbonic acids and chlorides. Their composition and the amount of water in the overhead stream will affect the pH and stability of the ferrous sulfide scale. This is directly related to the protective value obtainable from the sulfide scale and to severity of corrosion. Of course, gas velocity and turbulence also are significant factors.

The corrosion attack is in the form of general metal loss, pitting, hydrogen blistering or a combination of these. Corrosion rates on the shell may be as high as 15 to 25 mpy, on the condenser tubes 25 to 45 mpy and on the preheat exchanger in the vicinity of 5 to 10 mpy. Whether hydrogen blistering will occur depends on the sulfide scale stability and steel quality. Susceptibility can be detected readily with hydrogen probes. The phenomenon and damaging effects of low temperature hydrogen attack have been described.[7,8]

In addition, the acid injection point and area immediately downstream are

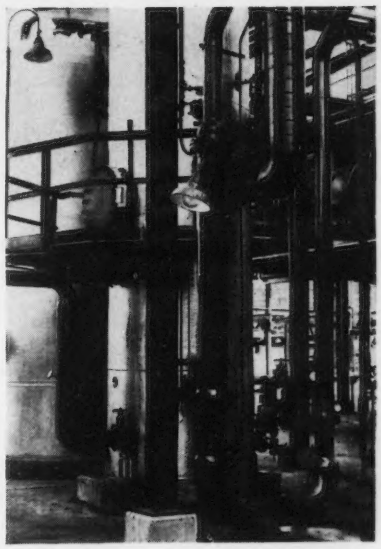

Figure 3—Duriron loop used for H_2SO_4 injection into sour water feed.

subject to sulfuric acid corrosion. This attack, which is separate from the sour water corrosion problem, can be solved with proper selection of construction materials. Hastelloy B or polytetrafluoroethylene are used generally for the nozzle where the concentrated acid, handled in carbon steel piping, is introduced into the sour water. High corrosion rates on other nozzle materials often have resulted from elevated temperatures caused by the heat liberated by H_2SO_4 dilution. For the area immediately downstream and for the mixing plates, Duriron has generally proved to be satisfactory. Figure 3 shows a Duriron loop used for H_2SO_4 injection into sour water feed.

Methods Used to Combat Corrosion

Several corrosion control procedures can be followed such as inhibition, nonmetallic coatings and alloy selection.

Inhibitors

The filming amine inhibitors in several cases have counteracted corrosion on carbon steel in the tower and overhead condenser. Close control, however, is important because in several instances these inhibitors were unable to provide an effective film on the steel probably because of turbulence and/or high velocity and low pH of the stripped vapors. This resulted in corrosion rates 2 or 3 times the normal rate; apparently the inhibitor's detergent action removed the partially protective corrosion scales. Effectiveness of inhibition may be determined from iron counts, corrosometer probes and hydrogen probes while operating, and from actual wall thickness measurements during shutdown. This follow-through evaluation is a desirable step.

Gunite and Other Non-Metallic Coatings

Several companies have adopted the practice of building sour water strippers with diameters large enough to allow for application of several inches of gunite at a later date if corrosion rates indicate

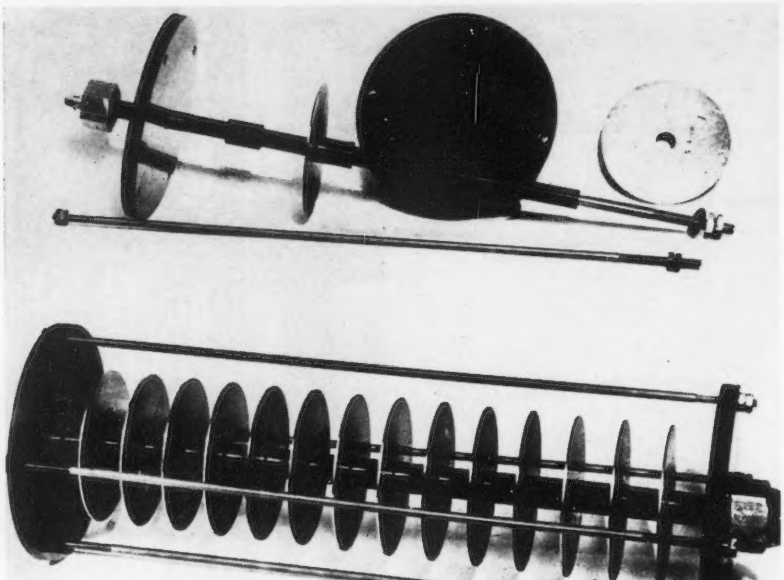

Figure 4—Test spools containing a variety of metal and alloy specimens used in exposure tests in units under service conditions.

the need. Also epoxy resin based paints have been used to protect the column wall and turbo-grid trays. These methods can be effective if the unit is inspected regularly and the coatings maintained in good condition.

Alloy Materials

Alloys can be justified frequently if corrosion rates on carbon steel exceed 25 mpy. Alloys are used for the heat-exchange tubes of the overhead condenser, as cladding or lining in the upper part of the tower and for the overhead transfer line.

In general, aluminum, Type 316 stainless steel and Monel (30-70 cupro-nickel) are the alloys to be considered for this system. They are almost equally resistant to wet hydrogen sulfide and wet carbon dioxide attack. Alloy selection depends on the individual system and on the concentrations of chlorides, cyanides, aliphatic acids, etc.

Aluminum generally is given first consideration because of its lower initial cost. Three points, however, should be kept in mind. First, if substantial quantities of chlorides and formic acid are stripped, rapid localized attack in the form of pitting will result. Second, copper salts contained in the system which may originate from corrosion of Admiralty bundles are detrimental to aluminum. Salts plating-out on the aluminum results in perforations. Third, aluminum heat exchanger tubes and carbon steel shells should be insulated from each other to prevent severe galvanic attack on the aluminum at points of contact. For the same reason, aluminum tube sheets should be used.

Type 316 stainless steel and Monel can handle a large variety of incidental corrodents which may be present in the sour water. The inclusion of molybdenum in the Type 316 alloy gives it fair resistance to small quantities of chlorides and pitting corrosion. Because stainless steels are susceptible to chloride stress cracking, failures may occur in stressed locations where tubes are rolled into tubesheets or adjacent to welds. When the chloride concentrations become substantial (for example when waste from the desalter units is included), Monel is the preferred alloy.

The Corrosion Engineering Section of the International Nickel Co. has cooperated with several refineries in plant corrosion tests to determine the most suitable construction materials for equipment used in the treating or handling of sour waters where these waters were the principal corrodents.

Test spools such as those shown in Figure 4 and containing a variety of different metal and alloy specimens were made available for exposure in operating units under actual service conditions. The specimens were carefully cleaned and weighed before and after test. From the weight losses based on duration of exposure, corrosion rates were determined in terms of inch penetration per year.

Field Experiences

Results of a plant corrosion test in the vapor phase of a water reclaimer tower are given in Table 1. The spool was exposed in the top head of the tower which was stripping contaminants from the stream wash waters before disposal. The pH of the tower feed was 7 to 7.5, the pH of the bottoms 9 to 9.5 and the condensate return from overhead was pH 8.2 to 8.7. Carbon steel was used originally for the tower trays and overhead condenser tubes. Satisfactory life was obtained from the trays. However, the 16

TABLE 1—Results of Plant Corrosion Test in Vapor Phase of Water Reclaimer Tower

Material	Indicated Corrosion Rate Inch Penetration Per Year
Monel	0.001
Inconel	<0.001
Types 302 & 304 SS	<0.001
Type 316 SS	<0.001
Type 410 SS	<0.001
Aluminum 2S	<0.001
Copper	0.011
Mild Steel	0.005
Ni-Resist	0.001
Cast Iron	0.004

Corrosive: Wash water vapor containing approx. 65-85 mol % H_2S, 7-12 mol % CO_2, 3-17 mol % NH_3 plus trace amounts of chlorides, cyanides and hydrocarbons.
Temperature: 250-265F (Ave. 260F).
Duration of Test: 388 days.

gage steel tubes in the condenser failed by general corrosion after only 2 to 3 months' service.

Data from the tests indicate a reasonably low and economic corrosion rate for carbon steel which is reflected in the good performance of the trays. The difference in performance between the trays and condenser tubes is due to the fact that in the latter instance the gas or vapors were cooled below the dew point and high corrosion rates were caused by the resulting acid condensate. Experience under these conditions has shown that Monel, Inconel and the 18-8 stainless steels are useful and highly satisfactory materials. Good experience also has resulted from the use of filming amine inhibitors in protecting carbon steel tubes by injecting amines into the overhead vapor line and returning them to the tower with the reflux.

In another case of field experience, Type 316 tubes in an overhead condenser failed in a stagnant area near the incoming vapor stream where the passivity was destroyed beneath water droplets. This resulted in a galvanic cell with the remainder of the tube. The same phenomenon occurred because of condensation against the tubesheet; failures occurred after 9 months' operation because of perforations where the tubes were rolled in. Figure 5 shows the type of failure experienced with the Type 316 stainless steel tubes.

Monel has performed well in this application and is generally considered to be a reliable material under variable or indefinite conditions where, because of the nature of the environments with respect to chlorides or the operation of the equipment, opportunities are provided for setting up of concentration cell corrosion and/or stress corrosion cracking.

Some references[9] are made about the good behavior of 90-10 and 70-30 cupronickels in resisting ammoniacal compounds such as occur in the feed-bottoms exchanger. However, where hydrogen sulfide concentrations are high as in the overhead system, attack is accelerated and high corrosion rates have been observed.

In Table 2, results are given of plant corrosion tests in a high pressure water absorption system for the removal of acidic gases such as CO_2 and H_2S from

hydrocarbons. This process was reported on by Bradley and Dunne[10] and included detailed corrosion measurements in the absorption pilot plant.

Test data indicate that all materials shown, with the exception of mild steel and cast iron, were highly resistant to general corrosion. However, Type 410 stainless steel and aluminum 2S were subject to rather significant pitting attack. Studies on the susceptibility of the materials to sulfide stress corrosion cracking indicated that they were free of deterioration by this phenomenon. But experience with pilot plant operation showed that with the use of hard materials such as ordinary spring steel for Bourdon tubes, relief and pump valve springs, sulfide corrosion cracking can occur.

Inhibition with a water dispersible, amine type inhibitor not only reduced the corrosion rate on carbon steel but also provided protection against sulfide stress corrosion cracking. Because the conclusions on inhibition were based on relatively short-time tests, the following corrosion control methods were proposed:

1. Pump casings, impellers and shafts —Type 304 or 316 stainless steel.

2. Shaft sleeves and casing rings—Stellite No. 1 over Type 316 stainless steel.

3. Impeller rings—K Monel.

4. Mechanical seal parts, rotating elements, locks and glands—Type 316 stainless steel.

5. Inserts—carbon with K-Monel springs.

6. Valve trim, thermometer wells and orifice plates—Type 316 stainless steel.

7. Bourdon tubes, relief valve springs —K Monel.

In some plants, excessive corrosion of carbon steel has been experienced in the stripping of hydrogen sulfide from diethanolamine-water solutions.[11,12] This problem has been overcome generally by use of filming amine type inhibitors. Data in Table 3 obtained from a plant corrosion test in water vapor containing diethanolamine, H_2S and traces of NH_3, CO_2 and hydrocarbons show that the corrosion rate of carbon steel can be negligible when inhibitors are used. The spool was installed in the top zone of an overhead regenerator accumulator, and the inhibitor was injected into the overhead vapor line. In addition to carbon steel, all other materials had insignificant corrosion rates with the exception of copper which suffered complete disintegration during the test period because of the ammonia or ammoniacal conditions present.

On occasion, sour waters contain from trace to significant amounts of chlorides, for example, when desalter water is added to the stream going to the stripper. This procedure can cause serious corrosion problems. The extent of damage might be illustrated by the data in Table 4, obtained from a plant corrosion test in a water separated from light straight run gasoline. This spool was immersed in the water phase at the bottom of a separator. Apparently, in addition to chlorides being present, there were small amounts of hydrochloric acid formed by hydrolysis of the chlorides.

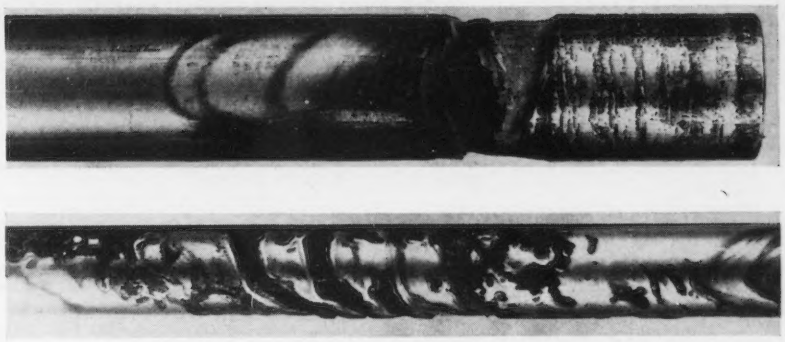

Figure 5—Type 316 stainless steel tube failure in a stagnant area near the incoming vapor stream of an overhead condenser. Failure occurred after a 9-month operation period.

TABLE 2—Results of Plant Corrosion Tests in Water Absorption of Acidic Gases From Hydrocarbons

Material	Indicated Corrosion Rate (ipy)	
	Top of Column	Bottom of Column
Monel	0.003	0.003
Inconel	0.0001	0.0001
Types 302 & 304 SS	0.0001	0.0001
Type 316	<0.0001	<0.0001
Type 410	0.004	0.004
Aluminum 2S	0.003	0.006
Mild Steel	0.034	0.038
Ni-Resist	0.015	0.012
Cast Iron	0.068	0.050

NOTE: All materials showed incipient pitting attack with exception of Type 410 and aluminum 2S which showed maximum pits of 0.008 inch and 0.003 inch respectively. Mild steel showed hydrogen blistering.
Corrosive: Absorbing Fluid—Water Feed Gas—32% H_2S, 12.5% CO_2 (by vol.)
Temperature: 90°—110F. Pressure: 500 psig.
Duration of Test: 23 days.

TABLE 3—Plant Corrosion Test in Stripping of H_2S From DEA Solution

Material	Indicated Corrosion Rate (ipy)
Monel	0.0035
Inconel	<0.0001
Type 302 & 304 SS	<0.0001
Type 410 SS	0.0002
Aluminum 2S	<0.0001
Copper	*
Mild Steel	0.0010
Ni-Resist	0.0006
Cast Iron	0.0010

* Specimens originally 0.031 inch thick completely destroyed during test period.
Corrosive: Vapors of water, DEA and H_2S containing traces of NH_3, CO_2 and hydrocarbons.
Temperature: 100F.
Duration of Test: 396 days.

TABLE 4—Plant Corrosion Test in Water Separated From Light Straight Run Gasoline

Material	Indicated Corrosion Rate (ipy)
Monel	0.0007
Inconel	<0.0001
Type 302 SS	<0.0001
Type 410 SS	0.012*
Aluminum 2S	0.006*
Copper	0.012*
Mild Steel	0.032**
Ni-Resist	0.004
Cast Iron	0.045

* Partly destroyed and perforated. Original thickness 0.031 inch.
** Partly destroyed and perforated. Original thickness 0.062 inch.
Corrosive: Separated water containing traces of gasoline, hydrogen chloride and 0.036% hydrogen sulfide.
Temperature: 61F.
Duration of Test: 266 days.

Type 410 stainless steel, aluminum 2S, copper and mild steel definitely are unsatisfactory for use under these conditions. Specimens of these materials were partly destroyed and perforated during the test. Although Type 302 stainless steel showed excellent resistance to corrosion in the test, austenitic materials generally are prone to localized attack in the form of pitting. Furthermore, under acidic conditions they are subject to stress corrosion cracking. Monel is a commonly used material under these conditions and probably is an economical solution. For cast equipment such as pump parts, valve bodies, etc., Ni-Resist has proved to be useful. As noted in the test data, it showed a superiority over cast iron of better than 10 to 1. With the development of the ductilized Ni-Resist with good thermal shock and corrosion resistance, this alloy may have wide acceptance in refinery service.

Phenolic wastes discharged from a refinery are another major source of stream pollution. Some plants have adopted the bacterial oxidation process to remove these wastes from the effluent; most refineries in areas where stream pollution is a problem are equipped with phenol recovery systems. A plant corrosion test was made in a recovery tower handling 10 percent phenolic water containing 50 to 1320 ppm of chlorides at 220 F. The spool was exposed in the vapor phase between the tower's first and second tray. Results are shown in Table 5. These data illustrate the strong effect of chlorides in

TABLE 5—Plant Corrosion Test in Phenol Recovery Tower

Material	Indicated Corrosion Rate (ipy)
Monel	0.004
Inconel	0.005
Types 302 & 304 SS	0.004*
Type 316 SS	0.004*
Type 410 SS	0.017
Aluminum 2S	**
Chemical Lead	0.012
Mild Steel	0.044

* Specimens suffered stress corrosion cracking.
** Completely destroyed—Original thickness 0.031 inch.
Corrosive: 10% phenolic water vapor containing 50-1320 ppm chlorides.
Temperature: 220F.
Duration of Test: 71 days.

causing stress corrosion cracking of austenitic stainless steels in this weak acid medium. Data prove that aluminum 2S is unsatisfactory in this type service: the specimens were completely destroyed during the test. Both Monel and Inconel are resistant to corrosion under these severe conditions.

Conclusions

1. As efforts are increased to eliminate water pollution, industry finds it difficult to predict whether or not serious corrosion will occur because of the large differences in contaminants and methods of treating water. Despite the fact that problems for each stripper are probably unique with no apparent correlation between systems, some general conclusions can be made on corrosion control of sour water stripping units.

2. For the overhead condenser, alloy materials such as aluminum, Monel and Type 316 stainless steel have effectively eliminated the main corrosion problem. Each alloy has certain peculiarities and shortcomings that should be considered in selection. These alloys have been shown to be justifiable where corrosion rates on carbon steel exceed 25 mpy.

3. Corrosion inhibitors, gunite and alloy lining have proved generally satisfactory in protecting the stripping column. The standard material of construction is carbon steel.

4. By improving plant practice (proper operation and control of the pH of the sour water feed), by avoiding the worst conditions (high temperatures and pressures, oxygen, velocity effects and impingment) and by proper selection of materials (Monel, Type 316. aluminum or gunite), good performance can be achieved.

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DISCUSSION

Discussion by A. J. Freedman, Standard Oil Company of Indiana, Whiting, Indiana:

Have you observed any cases of an increase in corrosion rate caused by inhibitors? If so, can you tell us what inhibitor concentration was used and what concentration was required to control the corrosion?

Reply by C. M. Schillmoller:

Increased corrosion caused by inhibitor addition has been observed both in the laboratory and in the field in cases where no protective scales or films are formed. This apparently can occur with certain inhibitors, at low concentrations say 1 to 2 ppm and also at concentrations of about 12 ppm where turbulence and velocity, or low pH conditions interfere with film formation. A great deal still remains to be learned about the nature of inhibitor action and the relationship between corrosion inhibition and detergency. Corrosion has been reduced substantially in a number of towers with 8 to 12 ppm inhibitor concentration.

Discussion by Donald L. Burns, Gulf Oil Corp., Port Arthur, Texas:

Have you encountered severe fouling of trays in the stripper with salts from the feed water such as $CaCO_3$? If so, have inhibitors with detergent action been of value or do you have a suggested remedy for this type fouling?

Reply by C. M. Schillmoller:

No, I have not observed such fouling in sour water strippers. As for the effectiveness of inhibitors to remedy this by their detergent action, I do not know. I wonder if one of the inhibitor suppliers would care to comment on this.

Discussion by D. R. Behn, National Aluminate Corp., Glendale, California:

Organic fiiming amine type inhibitors are not necessarily effective on calcium deposits. This problem can best be controlled by proper adjustment of pH to keep calcium in solution, reduction of calcium input to system, or by means of polyphosphate, organic dispersants of processed lignin type, or combinations of these chemicals and/or methods.

Discussion by R. B. Thompson, Universal Oil Products, Des Plaines, Illinois:

Is oxygen present in the overhead of sour water strippers and if so how much? It has been rumored that stainless steel tube bundles in distillation towers operating in an essentially oxygen free environment corroded very rapidly. The question then relates as to whether oxygen is present to passivate the stainless steel in the sour water strippers.

Reply by C. M. Schillmoller:

The units, I am familiar with, showed no or only minute quantities of oxygen in the overhead gas stream. Oxygen is not always required to maintain passivity. There are many instances in the chemical industry where Type 304 and especially Type 316 stainless steel maintain their passivity under reducing conditions if the corrosive environment is not too severe. For example: Stainless steels in low concentrations of sulfuric acid at moderate temperatures. However, the stainless steels may be subject to so-called border-line passivity effects under these conditions. This particularly shows in the presence of an electrolyte, where rapid localized corrosion may result, or uniform high corrosion rates of about the same order as carbon steel.

Any discussion of this article not published above will appear in the December, 1959 issue

Corrosion Protection Features Of the Hyperion Ocean Outfall*

By HARRY J. KEELING

Abstract

Details are presented on the planning, design and installation of corrosion protection facilities to provide 100-year design life for a 7-mile, 22-inch steel pipe sewer outfall, the outer end of which is in ocean water 340 feet deep. The outfall serves the sewerage systems of Los Angeles and 16 other adjacent municipalities.

An impressed current cathodic protection system is used to protect the exterior of the pipe which is wrapped with a multiple-layer coal tar coating reinforced with glass fiber with bonded impregnated asbestos felt shield. A reinforced pneumatically applied cement mortar jacket provides negative buoyancy. Interior is protected by centrifugally-spun mortar lining with epoxy sleeves at welded tie-in joints.

Because of the depth of water in which the pipe was to be placed several novel features were necessary. Cathodic protection was applied continuously during installation by the use of zinc anodes. This system was monitored to detect any serious coating holiday before the pipe was laid in deep water. Permanent test leads also were attached to the pipe so performance of the impressed current system could be checked, continuously.

After 1½ years' operation there has been very little change in effective coating resistance, a pipe potential of —1.05 volts versus a copper/copper sulfate electrode being maintained with consumption of about 150 ma.

5.2.3

About the Author

HARRY J. KEELING is a consulting engineer in Los Angeles and has been engaged in cathodic protection work since 1932. He was affiliated with Southern Counties Gas Company of California until he established his own consulting firm in 1953. He received a BS and MS from California Institute of Technology and has been a member of NACE since its formation.

Introduction

THE HYPERION Treatment Plant,[1] operated by the City of Los Angeles, California, disposes of sewage from the city proper and from sixteen surrounding municipalities. The welded steel ocean outfall completed in 1957 for discharging digested sludge from this plant is of interest to corrosion engineers for several reasons. The steel pipeline was designed to have a useful life of at least 100 years and the depth of the water makes it inaccessible for inspection or testing by usual methods. To date it is the deepest and longest pipeline of its kind in the world, extending seven miles into the open ocean with its end in water 340 feet deep.

Corrosion control features incorporated in this project to meet these unusual requirements may well have numerous applications to other off-shore pipelines which terminate in exceptionally deep and rough water inaccessible by conventional diving methods. This article will discuss briefly the steps leading to the selection of the final corrosion control design features incorporated in the specifications for construction and will review results obtained during the first 1½ years of operation of the line.

Feasibility Study

Over-all responsibility for the design of the Hyperion outfall was given by the city to Hyperion Engineers, Inc., a joint venture comprising the Los Angeles firms of Holmes and Narver, Inc.; Daniel, Mann, Johnson, and Mendenhall; and Koebig and Koebig. In 1955 during the preliminary design phase of the project a study[2] was made to evaluate the external corrosion hazard to the proposed pipeline and to develop the most satisfactory solution to the problem. The corrosion study and the criteria developed for the final design of the cathodic protection system were made on the basis of the following basic assumptions set up by Hyperion Engineers.

Assumptions

1. The desired design life for the pipeline was 100 years.

2. The pipe would be 22 inch o.d. API 5LX-52 steel, with 5/16-inch minimum wall thickness, a ½-inch cement mortar inner lining and a 1⅛-inch outer coat of reinforced mechanically applied cement mortar to provide negative buoyancy and physical protection during the installation phase.

3. The line would extend into the ocean for a distance of 6½ miles from shore, terminating in water 600 feet deep. (Actually the depth of the completed line is 340 feet, because an alternate longer route was selected.) Diving being impossible near the end at 350 to 600 foot depths, construction would be by welding the individual sections together at the shore end and pulling the sea end of the pipe into place with barges in one continuous pull.

4. It was anticipated that the pipeline would be trenched and buried to a depth of from 4 to 15 feet below the ocean floor for the first 6000 feet of off-shore section to protect it from heavy seas and surf. This would be done after completion of the line, by means of a device which would travel along the pipe carrying a series of jet nozzles to cut the trench.

Preliminary Design for Cathodic Protection

Two of the requirements listed for this job differ markedly from those of the usual cross-country pipeline or off-shore oil tanker loading line: The requirement for 100-year useful life and the inaccessibility of the off-shore portion of the pipe because of the depth of the water. These factors had the greatest influence in establishing the corrosion control system that was finally selected.

The cement mortar coating required to achieve the necessary negative buoyancy could be relied upon to provide corrosion protection to the steel provided it remained intact. However, because severe installation conditions were anticipated, it was not deemed advisable to assume that the pipeline could be pulled into final position without the possibility of either cracking the cement coating or losing perhaps several square feet if it should accidentally be subjected to extreme bending conditions or come into violent contact with unseen hard or rocky ledges in water too deep for diving operations.

Cathodic protection as an essential part of the corrosion control system seemed to be indicated, therefore, because it offered the only sure means of protecting the steel from corrosion at inaccessible points in case of failure of the external coating due to accidental damage during installation or in subsequent years. Damage to the line resulting from corrosion would be extremely costly, if not impossible, to repair because of the depth of the water. Thus the wisest course would be to prevent corrosion damage from occurring by incorporating adequate corrosion protection measures into the design.

Sacrificial Anodes

Cathodic protection of submarine pipelines has been successfully accomplished by attaching sacrificial anodes of zinc or magnesium directly to the pipe at several points along its length. This distributed anode system is a particularly desirable type of design for long pipelines, because it overcomes the inefficiency due to the

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attenuation effects of the longitudinal resistance of the pipe metal, and also because it eliminates the necessity for diving operations since the anodes usually can be attached to the pipe before it is submerged.

The use of sacrificial anodes distributed along the length of the pipeline could not be considered for this project for several reasons. In the first place zinc or magnesium anode installations could not be expected to last longer than 25 or 30 years without replacement or attention. Due to the depth of the water and the heavy seas in the off-shore sections, diving operations to locate and replace anodes mounted directly on the pipeline would be extremely difficult and prohibitively expensive at best, if not impossible. Anodes attached to the line in the inshore area probably would interfere with the travelling jetting machine used to bury the pipeline.

Impressed Current System

It thus appeared that for the proposed Hyperion outfall, a single installation draining current from the shore end would be the most desirable means of applying cathodic protection. An important advantage of using this type of system is that it facilitates determination of the total current demand by the inaccessible portions of the offshore pipeline. With appropriate testing facilities the operator can monitor continuously the condition of the coating, thereby having assurance that the line will not have to be shut down unexpectedly as a result of corrosion leaks. Thus he is given advance warning in case of trouble resulting from coating deterioration or damage and can take appropriate steps long before the corrosion of the steel has progressed sufficiently to perforate the wall of the pipe.

An impressed current system using a rectifier offered promise of the greatest flexibility, because it would be a rather simple matter to increase the current output should it be necessary, due to gradual deterioration or accidental damage to the coating during the useful life of the pipeline.

Current Requirements for Cathodic Protection

The next step in the preliminary study was to determine the current demand for a cathodic protection system as described above, with the current applied at the shore end only. In 1955 there was very little published information regarding the application of cathodic protection to cement mortar coated steel structures submerged in the sea. Most articles on this subject referred only in a general way to the fact that a coating of dense Portland cement mortar applied directly to a steel surface lessened the current requirement for protection, without giving specific information regarding actual current densities required. Preliminary computations[3] made it apparent that it probably would be impractical to try to produce sufficient current densities at the offshore end with a single rectifier connected to the inshore end if cement coating alone were used. Therefore, to insure successful operation, it would be advisable to decrease the attenuation effects on the pipeline by supplementing the cement mortar with an additional protective coating of higher dieletric value.

Computations were then made of current requirements for cathodic protection of the proposed pipeline, assuming various values of effective coating resistance (or conductance). These are summarized in Table 1, which shows the values of current and potential required at the shore end of the line to produce a change in (pipe-to-earth) potential of 0.2 volts at the extremity for various assumed values of effective coating resistance. For purposes of this study it was assumed that a change of 0.2 volts in (pipe-to-earth) potential at the sea end of the line would result in adequate cathodic protection to any portions of the steel surface that might be exposed through holidays or breaks in the coating. Because in an actual case it is impossible to know the exact area of steel exposed, for purpose of analysis it was assumed that the pinholes or breaks are distributed uniformly along the entire length of the pipeline. The effect of this is the same as if the coating itself had a uniform leakage conductance.

Table 1 shows that for a resistance of 4 kilohms (250 micromhos conductance) per square foot, 22 amperes at a potential difference of only 1.1 volt across the coating is required at the shore end to achieve adequate protection at the remote end. However, if the effective resistance is only 3 kilohms (333 micromhos conductance) per square foot, then a current of 38 amperes is necessary and the required potential at the shore end has to be increased to 1.5 volts. This value is higher than that recommended for bituminous coatings. For very conservative design it is considered advisable to restrict the voltage across bituminous coatings to about 1.1 volts in order to avoid all possibility of damage by electroendosmosis.

Thus it was found that a very satisfactory cathodic protection installation would be possible, provided the effective coating resistance was 4 kilohms per square foot or better. This value seemed well within the realm of possibility if sufficient care was given to the quality of the installation.

Summary of Feasibility Study

The corrosion study indicated that with careful attention given to design details it would be feasible to expect the inaccessible parts of the steel ocean outfall to be protected from corrosive attack by the sea throughout the 100 year design life of the project. Essential elements required to achieve this result were:

1. Selection of a protective coating system that could be expected to maintain a dielectric value of not less than 4 kilohms effective resistance per square foot throughout the life of the project.
2. Installation of an impressed current cathodic protection system at the shore end of the line.
3. Development of suitable techniques for testing the cathodic protection system that would give continual assurance that the system was providing satisfactory protection to the inaccessible portions of the pipe in deep water.

Only with a combination of materials, workmanship and inspection of the highest quality would it be possible to build a steel outfall that could be expected to remain satisfactory during a century of use under ocean environment. This fact was kept in mind constantly during the development of the design criteria discussed below.

Corrosion Protection Design Features

Coating

The protective coating for the exterior of the proposed steel pipeline should comply with the following requirements:

1. Must have a useful life of 100 years in a marine environment while subject to cathodic protection.
2. Must maintain a high dielectric value (at least 4 kilohms effective resistance per square foot) during the projected life of the pipeline.
3. Must resist the deteriorating effect of electrical current due to the cathodic protection installation.
4. Must withstand the physical stresses imposed by handling during construction, or by subsequent loads resulting from its location on the floor of the ocean. (The coating would only be required to withstand severe bending or shock stresses; adequate protection from damage due to abrasion, impact, or concentrated loading would be furnished by the reinforced cement mortar jacket used to provide negative buoyancy during the installation phase.)
5. Should have a cost as low as possible, consistent with the foregoing requirements.

In evaluating various coating systems for this particular project, polyvinyl chloride or polyethylene pressure sensitive tapes were considered because they offered great promise as a pipeline coating

TABLE 1—Current and Voltage Data for Cathodic Protection of a 22-Inch OD 5/16-Inch Wall Welded Steel Pipeline*

Coating Resistance, Kilohms/sq ft	Coating Conductance, Micromhos/sq ft	Impressed Current Shore End, Amps	Potential Drop Across Coating at Shore End, Volts
2	500	86.1	2.77
3	333	38.0	1.51
4	250	21.8	1.06
5	200	15.8	0.83
10	100	4.45	0.354
50	20	0.75	0.228
500	2	0.072	0.203

* Current impressed at shore end. Length = 36 kilofeet. Assumed potential drop across coating at offshore end of line = 0.2 volts.

Figure 1—Details of the cast epoxy sleeve used in place of cement mortar to line the field joints at the ends of the 600-foot strings. Interior of the pipe section on the right has been "buttered" with an epoxy adhesive and the joint is now ready for alignment with the clamps preparatory to welding.

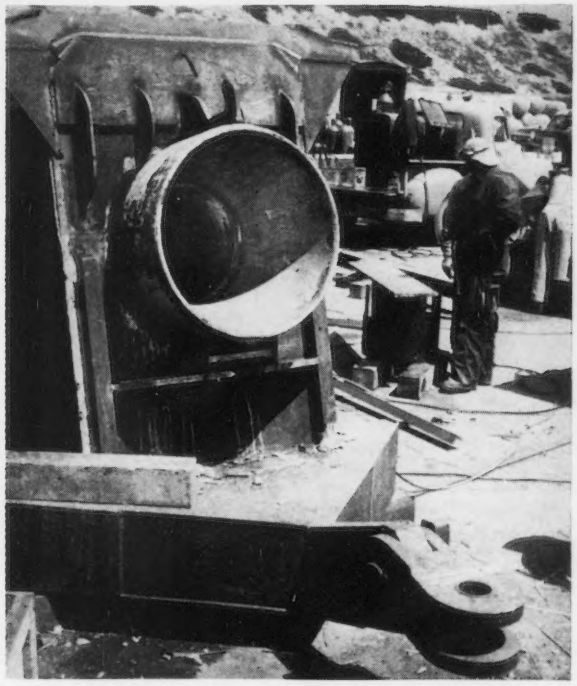

Figure 2—Partial view of the pulling sled under construction, showing details of the cast epoxy insulation on the divergent opening at the outfall terminus.

material, because polyvinyl chloride and polyethylene have excellent dielectric characteristics and resistance to water absorption. However, in 1955 they had not been in general use long enough to provide an experience record that would justify their use in a project for which a 100 year life is a requirement.

Both asphalt and coal-tar products have been used successfully as dielectric membranes on cathodically protected submerged pipelines. Of the bituminous coatings, past experience records then available indicated that the most successful performance on submarine pipelines had been with either coal-tar enamel, or the mastic type asphalt reinforced with sand or other inert materials.

The coating system included in the final specifications embodied the following features:

1. Three coats of coal-tar enamel, total thickness 7/32-inch, and two layers of fiber glass reinforcing, with bonded asbestos felt (not over 25 percent saturation) outer wrapper.
2. Cement-mortar shield, mechanically applied, 1⅛ inches thick, reinforced with hot-dipped galvanized steel mesh. Mortar mix to be designed to achieve a high density coating with low moisture absorption characteristics.
3. Coating on the field joints to follow the same construction as used on the steel pipe.
4. A ½-inch centrifugally spun cement mortar interior lining.

Field Joints

The beach at the construction zone is only 600 feet wide between the surf line and a high bluff, therefore, to reduce the time required to pull the line into place, a 1000-foot launching pier was built, extending approximately 650 feet beyond the surf line, so that two 600-foot strings could be jointed simultaneously and the pull made in 1200-foot increments. During this operation the pipe was supported on pneumatic rubber-tired wheels, spaced at close enough intervals on the launchway to protect the pipe coating from damage from concentrated loading or excessive bending stresses.

The installation process, which has been described by others,[4,5,6] was not started until all 61 separate strings, each approximately 600 feet long, were made up and ready on the beach. In making up these strings, the coated and lined pipe sections from the mill were field welded at the site. Joints of the interior lining of each section in the 600-foot strings were made with cement mortar centrifugally applied by machine. Slings were used to apply coal-tar coating to exterior joints, following the same general specifications used for the mill wrapped pipe. The coating was then checked with a holiday detector and the galvanized steel reinforcing mesh applied. The cement mortar jacket on all field joints was pneumatically applied.

Epoxy Liner

Special procedures were developed to protect interiors of field joints connecting the 600-foot strings during the pulling operation. If conventional methods had been used, it would have been necessary for a man to crawl 1200 feet inside the pipe to complete the interior mortar joints, so protection of the unlined pipe at these field joints was provided by ½-inch thick pre-cast epoxy sleeves, as shown in Figure 1. These were cemented into place with epoxy resin adhesive to fill all voids between the ring and the steel pipe or the cement mortar lining. In developing this technique tests were run to determine if the heat of welding would be detrimental to the epoxy. It was found that the weld could be made immediately after putting the epoxy sleeve in place without any appreciable effect on the interior plastic cylinder.

It was found by using a suitable mix of high early-strength cement mortar, the joint could be pulled into the water immediately without damage provided it was covered with a sheet of steel held in place with bands. This steel sheet protected the fresh cement mortar from damage in passing over the launching rollers and from erosion due to wave action while it was entering the water.

Insulation

A review of the experience of operators of shorter cathodically protected offshore petroleum products loading lines disclosed that there usually was considerable current pickup from the uninsulated anchors attached to hold the line in place, and particularly from the pulling sled structure at the offshore terminal of the line. A considerable portion of the protective coating on such sleds was usually lost by abrasion by the time the line was finally pulled into position. Although the extra current required as a result of these holidays in the coating might be of little consequence on shorter lines, because of its length and electrical attenuation characteristics, the exposure of several square feet of bare metal near the terminal of

the proposed Hyperion outfall could not be tolerated. Therefore the pulling sled required to guide the pipe along the bottom during the pulling operations was specifically designed to transfer a pulling load of 200 tons to the end of the pipe without damaging the dielectric value of the protective coating.

The load from the pulling line attached to the sled was transferred through reinforced concrete in compression to suitable rings welded to the exterior of the pipe. Before placing the concrete in the welded steel sled structure, all portions of the pipe and its anchor rings were carefully coated with the glass-fiber reinforced coal-tar coating specified for the remainder of the line. After electrical inspection with a holiday detector, the pulling sled was filled with a concrete having a very rich mix to achieve high compressive strength and low water permeability.

An epoxy resin interior lining extending several pipe diameters from the discharge opening was used instead of cement mortar at the terminus of the outfall. This material was selected because it would resist the abrasive action of sand or solid particles in the zone of turbulence from the sludge stream and at the same time provide good electrical resistance. All parts of the steel divergent discharge nozzle, shown in the photograph, Figure 2, were encased in cast epoxy resin. This exterior epoxy end cap was carried far enough back into the concrete of the sled structure to anchor it securely and provide a long path for current leakage.

Submarine Test Cable

For maintenance or adjustment purposes it is customary to measure the potential at the remote end of shorter cathodically protected marine loading lines from a small boat, either through permanent test leads connected to a telephone buoy, or by temporary leads connected to the line by a diver. This method could not be used for the Hyperion outfall, because the terminus would be in such deep water that it would not be economically feasible to provide or maintain such leads. Marine loading line operators had reported that flexible insulated leads terminating in telephone buoys required frequent repairs or replacement because of damage from constant flexing as a result of wave action.

Therefore, specifications for the Hyperion outfall included provisions for incorporating a multi-conductor insulated submarine test cable into the construction of the pipeline, so that potential readings of the offshore portion of the line could be made from the onshore end to determine whether adequate pipe-to-earth potentials were being maintained. Minimum requirements for testing a cathodic protection system on an offshore pipeline would be achieved by connecting an insulated test lead to a point near the remote end and running it to shore. However, because additional conductors could be provided at relatively small incremental expense, provision was made in the specifications for inclusion of spare conductors in the system to provide for unforeseen contingencies which might result in the loss of one or more conductors during installation or in subsequent years.

The offshore portion of the pipe was divided roughly into thirds and a pair of test leads was installed in each. Individual conductors in each pair were connected to the pipe at points approximately 200 feet apart for several reasons: By the 200-foot separation it was hoped that one of the leads would survive if an accident destroyed the other. The use of duplicate leads going to approximately the same place on the pipe would give more reliability and credence to the test readings, and the separation also would permit the test engineer at the shore end to measure the potential drop across the 200-foot span of pipe and thereby determine the current flow in the line at that point.

In laying out the arrangement for the test leads, consideration was given to the steep grade near the outer terminal of the pipe. The final profile indicated that while the outlet of the pipeline would be in water between 320 and 340 feet deep, at a point 600 feet inshore from the terminal the water would be only 250 feet deep. Therefore the first pair of test leads was placed at this nominal location, which was 36 kilofeet from shore. The second pair was placed at 24.5 kilofeet from shore in water 160 feet deep and the third pair at a point 14 kilofeet from shore in water 110 feet deep. Plans were drawn to show these nominal locations, but exact locations were determined during construction by placing the connection to the pipe in the nearest adjacent field joint in order to minimize damage to the pipeline coating.

Submarine cables for the six offshore stations were multiple conductor cables, each conductor consisting of size 14 AWG, 7-strand cadmium bronze, with a minimum insulation of 5/64-inch rubber and 3/64-inch neoprene jacket. Consideration was given to the use of a steel armor on the test cables in order to give them more strength and resistance to abrasion. This idea was rejected, however, when it was found by theoretical analysis that the steel armor on the submarine test cable would constitute a serious threat to the proper operation of the protection system, and make it impossible to maintain the pipe at adequate potential. Therefore, only rubber and neoprene were used as outer armor for the cables. This type of construction, while more susceptible to damage from dragging ships' anchors, etc., would never be likely to interfere seriously with the protection of the line.

A six-conductor cable was used from the shore to the first test box located 14 kilofeet out, where one conductor was attached to the pipe. The cable was carried through the first box approximately 200 feet along the pipeline to the second box, where the second conductor was attached to the pipe and the remaining four spliced to a four-conductor cable. This general arrangement was followed until the last box was reached with a two-conductor cable.

The splice boxes, which were used also to insulate the conductor brazed to the pipe, consisted of 8-inch diameter threaded pipe nipples, 4 inches long, trimmed to fit the pipe and welded to it 45 degrees down from the top. This position was selected to prevent interference with the travelling jetting machine used to bury the 6000-foot offshore portion of the pipeline. Figure 3 shows how the cable entered the splice box through a waterproof U. S. Navy style compression sealing packing gland attached to the end of pipe nipples welded into the wall of the splice box. The conductor terminating in the splice box was welded to the pipe, and after screwing the threaded cover into place the splice box was filled completely with epoxy resin for insulation (Figure 4). The splices between the the multi-conductor cables were insulated individually with rubber splicing compound and tape before being encased in the epoxy-filled splice box.

The cable was supported on the pipe at intervals of 20 feet by means of wood blocks and steel banding passed around the pipe over the mortar coating. These temporary supports were required only to keep the cable in position during the installation period, because it was expected that soon after installation, bottom conditions in the offshore areas would cause the pipe and cable to be buried in sand. Adjacent to the splice boxes, strain relief was provided by pairs of micarta clamping blocks which were held in position by similar Monel bands (Figure 3).

Cathodic Protection During Installation

Because of the method used for constructing the line and the anticipated difficulty of correcting damage to the coating in such deep water, the specifications called for continuous testing of the coating system during the installation period through the application of cathodic protection. This would result in rapid polarization of any exposed steel at holidays and would give continuous inspection of coating quality and condition of the test leads. A zinc sacrificial anode system was specified as the most convenient method of applying cathodic protection during construction.

Zinc was selected in preference to magnesium, because its lower solution potential (—1.1 volt referred to $CuSO_4$ electrode) matches closely the optimum value desired for the pipe-to-earth potential at the shore end with the entire outfall line in place and the permanent cathodic protection station working. To take advantage of the favorable current regulating characteristics of zinc, circuit resistance of the temporary cathodic protection system was kept low by wiring of ample size and by using a sufficient number of long zinc anodes.

The zinc anodes were suspended from the launchway pier near the outer end and attached to an insulated collector cable supported by the launchway and terminating in an instrument shack on the beach near the water's edge. From the instrument shack the anode circuit extended through long flexible leads terminating in clamps which could be attached to the pipeline. Duplicate clamps

and leads were provided so that there would always be at least one connection to the pipe on the launchway. During the period while the two 600-foot strings were being jointed on the launchway, the clamp would be attached at Weld Station No. 1, which was near the offshore end of the pier. Just before the beginning of the 1200-foot pull, the second clamp was attached to the shore end of the moving pipe section and the forward clamp removed. As the pipe travelled down the launchway, the flexible cable attached to the clamp permitted application of current from the zinc anodes. Figure 6 shows a workman guiding the cable and test leads during this operation.

Both of the clamps also were equipped with insulated wire of smaller gauge which was connected to a recording potentiometer located in the instrument shack. This gave a continuous record of pipe-to-earth potential as measured with reference to a reference electrode installed at the water's edge. A recording millivolt meter in conjunction with a multirange shunt was used to record the current flow from the pipe through the zinc anode system.

The spool holding the multi-conductor insulated test cable used for the permanent test leads on the pipeline was placed near the instrument shack, so that the shore end of the cable would be convenient for testing.

Coating Tests During Installation

Primary purpose of the instrumentation and sacrificial anode system used for cathodic protection during the pulling operation was to give inspectors an immediate indication if a defective section of pipe hit the water. Any appreciable area of bare metal or large holiday would be disclosed by the sudden increase in current, or by the sudden fall in potential of the outer end of the pipe, as measured through the multiconductor test cable. If necessary, the pulling operation could be stopped instantly until the cause of the trouble was determined and appropriate steps taken to correct it. By this means it was hoped to discover defects in the pipe coating at the water's edge, where repair could be made more easily than if the defect was discovered after the pipe was in 50 feet or more of water. Due to careful workmanship and thorough inspection, not once during the seven and a half day pulling operation was it necessary to stop or delay pulling because of defective coating.

Rectifier Installation

The permanent cathodic protection installation consists of a 24-volt 50-ampere air-cooled unit of the selenium type, located in the electrical room of the Hyperion Plant. The ground bed consists of 8 high silicon cast iron anodes located adjacent to a concrete storm drain outlet structure on the beach, approximately 600 feet from the pipeline. The anodes were buried in the surf zone a minimum of two feet below mean low water elevation and at least four feet below the prevailing grade. The rectifier and ground bed have ample reserve capacity not only to take care of the ocean outfall but also to provide cathodic protection to some of the plant piping.

Figure 3—View of the cable splicers taken at 3 am, with the pipe in position on the launchway. The man in the foreground is installing the packing which forms a waterproof seal between the multi-conductor test cable and the splice box. Directly to his right is one of the micarta strain relief clamping blocks held in place by Monel metal bands.

Testing Facilities

Permanent wiring attached to the onshore piping at several points, together with the submarine multi-conductor test cable attached to the offshore portions of the pipe, terminate at a test panel located at the Hyperion Plant. These facilities permit making a potential survey from that point to insure that the pipe is receiving adequate cathodic protection.

Buried insulated conductors also were installed to permit testing the insulation of the several casings between the beach and the plant and to read the potentials of zinc and rusty iron reference electrodes which were installed adjacent to the pipeline at several locations on the onshore section. Although a copper-sulfate reference electrode placed over the pipeline at the water's edge is the basis for establishing the correct pipe-to-soil potential for the system, the installation of permanent reference electrodes greatly simplifies making routine tests.

Summary and Conclusions

Performance of Test Leads

Operation of the submarine test leads was completely satisfactory during installation of the offshore pipeline, until about five miles of the outfall was in place. At that point in the operation there were indications that several conductors in the cable had become defective. Damage to the cable insulation was not sufficient to increase the current demand by the pipeline appreciably, so no attempt was made to locate or repair the trouble until installation of the line had been completed. Sufficient good conductors remained in satisfactory operating condition to permit surveillance of the pipe coating.

Figure 4—Showing one of the completed test boxes being filled with epoxy resin after attaching submarine test lead to pipe. The test box, which has been primed ready for application of coal tar, is installed at one of the field joints in the 600-foot pipe strings.

The cause of the injury to the cable has never been established. There is a possibility that the damage was caused by anchors from small fishing boats that were active in the area during the time that the line was being pulled into place. After preliminary tests, the location of the defective portions was determined from the shore end by means of an oscilloscope and a pulse generator. One bad spot was discovered in relatively shallow water, so that it could be cut and the ends brought to the surface where the damaged portion was removed and a new section spliced in.

Further tests disclosed that the two-conductor cable had developed an "open" close to the onshore test box of the pair located near the extreme outer end of the outfall. Because of the difficulty of establishing the exact location and nature of the fault, the difficulty of making any repairs in 260 feet of water and the possibility of causing damage to the pipe coating in attempting to make repairs, it was decided to abandon the outer pair of test boxes.

Adequate surveillance of the operation of the cathodic protection system can be made with the four remaining test conductors, which have performed satisfactorily during their first 1½ years of operation and have shown no sign of further deterioration since repair was made to the damaged insulation.

Figure 5—Application of glass fiber mesh and hot coal-tar coating to the test box shown in Figure 4. The rubber insulation adjacent to the strain relief clamp has been wrapped with asbestos yarn to protect it from the hot coating material.

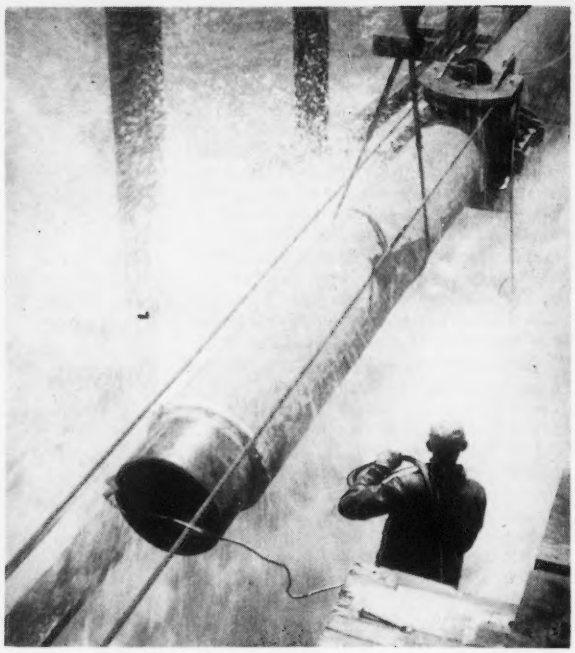

Figure 6—A wave breaks over the pipe travelling down the launchways during a "pull." The workman is walking beside the rear end of the pipe holding the slack ends of the submarine test cable and the cathodic protection wires leading to the clamp attached to the pipe. The hold-back clamp for the moving string of pipe can be seen in the upper right hand corner.

Performance of Coating and Insulation

The coating system has performed remarkably well. The entire line was installed without a single detectable coating defect. The effective coating resistance was approximately 400 kilohms per square foot; this was much higher than anticipated by the designers. During the first 1½ years of operation of the outfall, there has been very little change in effective coating resistance.

Immediately following completion of the offshore portion of the line on May 28, 1957, a pipe-to-copper sulfate reference electrode potential of —1.02 volts was maintained with a total current of 50 milliamperes drained by the zinc sacrificial anode system. By July 10, 1957, the pipe-to-soil potential was —1.05 volts and the current 30 milliamperes. These typical values held throughout the construction period while the shore end of the line was being tied in to the plant piping and the construction of the permanent cathodic protection installation was being completed.

Early in October, 1957, the current requirements for the pipeline, which was now tied in to the plant and discharging sludge, suddenly increased to nearly 4 amperes to maintain adequate pipe-to-soil potential. Investigation revealed that the insulated coupling installed at the onshore end of the steel outfall where it was jointed to the cast-iron piping leading in to the plant had unaccountably become short circuited. Because the coupling was buried about 15 feet deep in a rather inaccessible location, it was decided to abandon it and insulation of the outfall line was provided by cutting out a 40-inch section of cast-iron pipe on the inside of the plant building. This section was replaced by a similar length of 18-inch diameter cement asbestos pipe installed between couplings. The current demand on the steel outfall line then dropped to about 150 milliamperes to maintain a pipe-to-soil potential of —1.05 volts. Of this total current, line drop tests showed that approximately 50 milliamperes was being collected from the offshore portion of the line, and about 100 milliamperes from the 25-foot section of bare cast-iron pipe between the foundation wall of the building and the defective insulator. Tests made nearly a year later in December, 1958 showed essentially the same results.

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Any discussion of this article not published above will appear in the December, 1959 issue

The Classical Potentiostat: Its Application To the Study of Passivity*

By N. D. GREENE

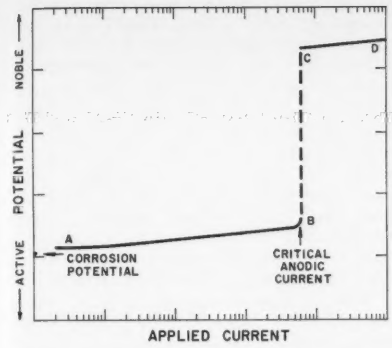

Figure 1—Anodic polarization curve obtained by varying current.

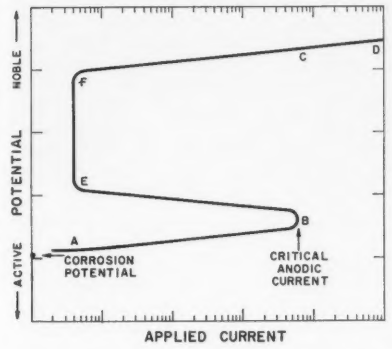

Figure 2—Anodic polarization curve obtained by varying potential.

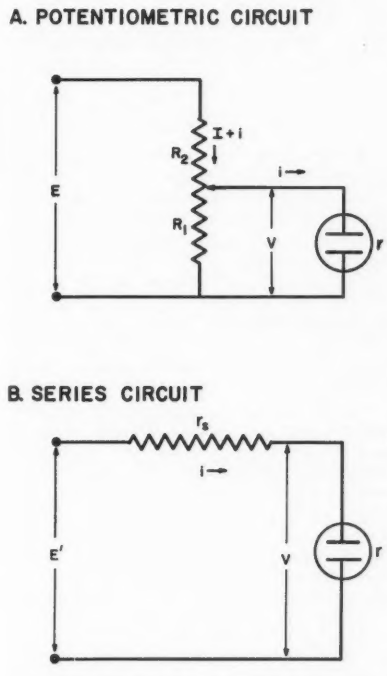

Figure 3—Comparison of potentiometric and series circuits.

Introduction

THE STUDY of passivity phenomena has been hampered because the passive state could not be adequately defined or characterized. A metal in a given environment could be described only as active or passive, depending on the observed corrosion rate and potential, with no clear division between these states. However, recent polarization measurements using potentiostatic techniques have shown that it is possible to quantitatively characterize active-passive systems.[1-7]

A potentiostat is a device which maintains an electrode at any desired potential. The literature contains a complex array of potentiostatic devices. The classical potentiostat, a term coined by Prazàk[8] is a simple, manually operated instrument which can be readily adapted to studies of passivity. Although the classical potentiostat may appear rather simple in comparison to electronic instruments specifically designed for passivity studies,[8-15] it nevertheless possesses the desirable combination of low cost, high accuracy, and circuit simplicity.

This paper develops the principles of the classical potentiostat and describes their application to the design of an instrument suitable for precise electrochemical studies of passivity.

Electrochemical Behavior of Passive Metals

The electrochemical characteristics of metals demonstrating passive behavior have been described in detail elsewhere[1-7] and need only be discussed in part here.

If a metal demonstrating typical active-passive behavior is anodically polarized by a constant current source, a curve similar to Figure 1 is obtained. The metal in the active state corrodes at potential A. At small impressed current densities, normal anodic polarization behavior is observed; the dissolution rate increases and the potential becomes more noble. When some current, such as B, is reached, the potential changes spontaneously to a considerably more noble value and oxygen is usually evolved. Current B is the critical anodic current for passivity. The principal disadvantage of polarization data obtained in this fashion is that the passivation reactions occurring in region BC cannot be examined in detail.

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Abstract

The potentiostat, a device which maintains an electrode at a constant potential, offers a unique means of characterizing the active-passive behavior of metals. The classical potentiostat possesses the desirable combination of low cost, high accuracy, and circuit simplicity. The basic principles of this instrument have been developed and applied to the design of an instrument suitable for electrochemical studies of passivity.

A quantitative discussion of the classical potentiostat shows that the total series resistance and the maximum current output completely characterize the performance of this instrument. Its performance can be increased by reducing its total series resistance and maximum current output. Both quantities can be experimentally determined.

An improved classical potentiostat with an ultimate potential control of ± 1 millivolt is described in detail. 2.4.2

If the system illustrated in Figure 1 is anodically polarized by varying potential rather than current density, the schematic polarization curve of Figure 2 is obtained. Logarithmic scales are used because of the range of current densities encountered. Active and passive dissolution rates often differ by factors of 10^5 or more.[1, 4, 6]

The inner loop BEFC of Figure 2 is of prime importance in characterizing passive behavior. Potential B, difficult to measure by constant current methods,

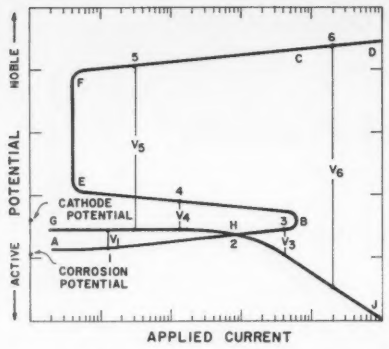

Figure 4—Potentiostatic polarization measurement.

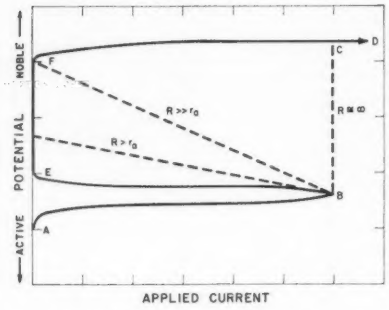

Figure 5—Influence of series resistance on measured anodic polarization curve.

signifies the first appearance of the passive state. E is the minimum potential for stable passivity in many systems. The current corresponding to line EF is the passive dissolution rate while the length of this line indicates the range of stable passivity. At F, the transpassive region begins, the metal dissolution rate increases, and oxygen evolution occurs. Experimental curves similar to Figure 2 can be easily measured by potentiostatic techniques, which accounts for the current interest in these methods for the study of passivity phenomena.

Theory of the Classical Potentiostat

General Principles

A constant potential source provides a potential which is essentially independent of current drawn from it. An obvious source is a battery or storage cell with large capacity. However, only certain voltages and voltage multiples are available from storage cells. To provide a continuously variable potential source, a voltage divider may be employed as shown in Figure 3A. Here, E is the battery potential, R_1, and R_2 the resistances of the potential divider legs, I the current through the inner loop of the potential divider, i the current output, V the potential output, and r the resistance of an electrolytic cell (or any load resistance).

The circuit shown in Figure 3A can be represented by the equivalent series circuit illustrated in 3B by application of Thévenin's method.[16, 17, 18]

$$E' = \frac{R_1 + R_2}{ER_1} \quad (1)$$

$$r_s = \frac{R_1 R_2}{R_1 + R_2} \quad (2)$$

E′ is the equivalent series potential and r_s is the equivalent series circuit resistance. Figures 3A and 3B are equivalent; the current output i and the potential drop V across the cell resistance r have the same values in both circuits if Equations (1) and (2) are satisfied.[1]

In actual potentiometric circuits there are resistances other than the voltage divider. An ammeter or precision resistor is required to measure the output current i. The connecting wires and other parts of the output circuit contribute additional series resistance. Thus, the total series resistance of a potentiometric circuit may be represented by:

$$R_p = r_s + r_i + r_m \quad (3)$$

where R_p is the total series resistance of the circuit,[2] r_s the equivalent series resistance of the voltage divider network, r_i the resistance of the ammeter, and r_m miscellaneous resistances of lead wires and contacts in the circuit.

The electrical characteristics of a potentiometric or voltage divider circuit are directly related to the relative magnitudes of its total series resistance R_p and its cell or load resistance r. When the resistance values are such that $R_p >> r$, the circuit acts as a constant current source. Under these conditions the current i is controlled mainly by R_p; changes in r have only a negligible effect on the magnitude of i. When $R_p << r$, the potentiometric circuit exhibits constant potential behavior. The current output is now primarily controlled by r. Although a change in r has a pronounced effect on current output, it has only a slight effect on V. For a given value of R_p, the decrease in V at any value of i is given by Ohm's law; $\Delta V = R_p i$. Thus, the potential output becomes more nearly constant as R_p and i are reduced.

From the preceding discussion, it is apparent that there is no sharp division between constant current and constant potential sources, but rather a continuous gradation between these two types. The quantitative behavior of a source can be completely described by its total series resistance R_p and its maximum output current i.

Potentiostatic Polarization Measurement

Potentiostatic measurements of anodic polarization may be accomplished by use of a constant potential potentiometric circuit such as described above. Referring to Figure 3A, consider that the load consists of a cell containing a corroding metal electrode and an inert cathode. Figure 4 illustrates a method for obtaining the anodic polarization curve for the corroding metal. Curve ABEFCD is the anodic polarization curve for the test electrode, and GHJ the polarization curve for the inert cathode. The anodic curve is obtained by applying various constant potentials (V_1, V_2 (equal to zero), V_3, etc.) across the cell and measuring the corresponding currents and anode potentials (points 1, 2, 3, etc.). Note that up to point 2 on the anodic polarization curve, the constant potential source is connected in opposition to the EMF developed by the cell. After point 2, the source is connected in series with the cell EMF. Switching at point 2 may be avoided by using a potential divider circuit with a zero-point center as employed by Bartlett.[19, 20]

The anodic polarization curve of Figure 4 is accurately measured only if the anode can be maintained at any desired potential. The use of a constant potential source does not ensure that the anode potential will be constant. This can be shown by examining the resistance sources within the electrolytic cell. Since the slope of a polarization curve has the dimensions of resistance, anodic and cathodic polarization may be represented by appropriate resistances. Thus, the total resistance, r, of the cell is given by:

$$r = r_a + r_c + r_e \quad (4)$$

Here r_a and r_c are the resistances arising from anodic and cathodic polarization, respectively, and r_e the resistance of the electrolyte. For most real systems r_a and r_c are not constant, but vary in a complex manner with current. This aspect is discussed further below.

In a potentiostatic measurement it is desired to measure current through r_a as a function of the potential drop across it. The resistances contributed by polarization of the cathode and by the electrolyte act as series resistances. Therefore, the total series resistance, R, of a potentiostatic circuit is given by:

$$R = r_s + r_i + r_m + r_c + r_e = R_p + r_c + r_e \quad (5)[3]$$

Figure 5 shows the effect of the total potentiostatic series resistance R, on the measured anodic polarization curve for the system illustrated in Figure 2. Linear scales are used to simplify the discussion of resistance effects. When R is extremely large, a constant current circuit is obtained. The polarization curve is similar to that of Figure 1. As the value of R is decreased, the measured curve approximates more closely the complete curve ABEFCD, and greater portions of the inner loop BEFC are observable. The dotted line portions in Figure 5 represent conditions under which it is usually impossible to maintain the anode potential constant with a classical potentiostat. The electrode potentials rise spontaneously and rapidly along these lines during anodic polarization. As pointed out by Bartlett[19] and Pražàk,[8] the slopes of these lines are equal to the total poten-

[1] All the calculations in this paper are based on the assumption that the battery potential E is a perfectly constant potential source. This assumption is closely approximated in actual circuits by the use of large-capacity batteries.

[2] It is important to note that R_p does not include the load or cell resistance r.

[3] Here, as in the case of Equation (3), R does not include the resistance, r_a, across which the potential is measured. For potentiostatic cathodic polarization measurements, Equation (5) reads: $R = R_p + r_a + r_e$

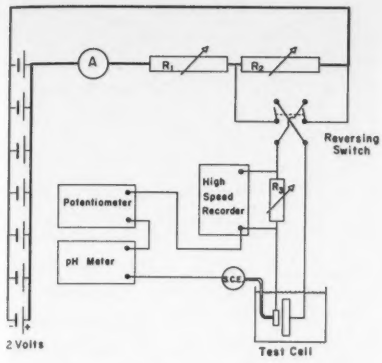

Figure 6—Classical potentiostat circuit diagram.

tiostatic series resistance R. The complete anodic polarization curve ABEFCD can only be measured if R is equal to, or less than the minimum slope of BE.

From Equation (5) and Figure 5 it can be seen that even though R_p may be small, resistance contributed by r_c and r_e could make the total circuit resistance very large. Thus, polarizing with a constant potential source does not always yield an accurate anodic polarization curve. Since r_e and r_c can have a pronounced effect on performance, the cell should be considered as an integral part of a classical potentiostat. The behavior of a classical potentiostat is quantitatively described by its total series resistance R, and its output current i. Reducing one or both of these parameters improves potentiostat performance.

Design of a Classical Potentiostat

Apparatus

The potentiostatic apparatus shown in Figures 6, 7, and 8 has been designed to minimize total series resistance R and output current i. The circuit, shown in Figure 6, is a modification of that used by Francis and Colner[21] for electropolishing experiments. Three 153 ampere-hour, 6 volt, automotive storage batteries with their 2 volt cells in parallel serve as a high capacity, constant potential source. Resistances R_1 and R_2, 1000 or 1500-watt compression carbon rheostats (James G. Biddle Company, Philadelphia, Pennsylvania), control the inner loop current I.(4) The reversing switch arrangement permits smaller values of R_1 and R_2 than would a zero-center circuit.[19,20] Current output, I, is measured with a 2-50 millivolt, adjustable span, Leeds and Northrup recorder in conjunction with a 0.01 to 11,111.1 ohm, precision decade resistance box (R_3). Electrode potentials are measured relative to a saturated calomel electrode (S.C.E.) using a Beckman Model G pH meter as a null indicator.

(4) Compression carbon rheostats, although possessing the desirable characteristics of high-current capacity and freedom from thermal drifting, produce noisy DC outputs. However, this noise is undetectable in anodic polarization measurements except for small, random current pulses which sometimes appear in the passive region.

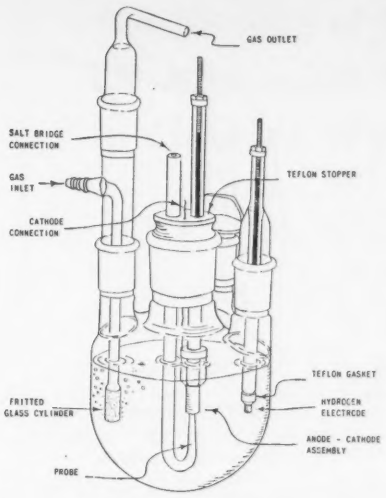

Figure 7—Test cell.

The test cell, shown in Figure 7, is a four neck 1000-ml flask. Compression Teflon electrode seals are used to prevent leakage and crevice attack. Teflon stopper allows adjustment of the relative positions of the probe and specimen holder. Test cell atmosphere may be controlled and a condenser attached to the extra neck permits experiments at temperatures up to the boiling point.

The anode-cathode assembly, shown in Figure 8, utilizes a large low over-voltage cathode and a small anode-cathode separation to minimize the values of r_c and r_e. The cathode is a platinized platinum tube, 1 x 2 cm long, which is positioned by a Teflon slip ring. A small anode area reduces the required output current i. Standard anode specimens are drilled and tapped cylinders 0.48 x 0.64 cm long, although smaller specimens may be used to further reduce the output current.

Performance

The total series resistance of the apparatus described above was measured to determine its potentiostatic performance. R_p was determined by measuring the potential output V as a function of output current i and applying Ohm's law. R_p and its component resistances are illustrated in Figure 9. It is interesting to note that r_s varies with output potential according to the parabolic equation:

$$r_s = [0.25 - \left(\frac{V}{E} - 0.5\right)^2][R_1 + R_2] \quad (6)$$

The short leads of the circuit were effective in maintaining r_m at a negligible value.

Using a platinized platinum anode and $0.01\overline{N}$ potassium chloride solution, the cell constant of the test cell was determined by an alternating current conductivity measurement.

The electrolyte resistance, r_e, for any solution may be calculated from this cell constant and specific conductivity data. Typical values for sulfuric acid solutions are given at top of column to the right:

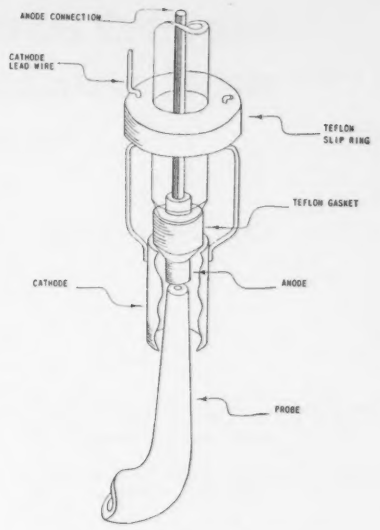

Figure 8—Anode-cathode assembly.

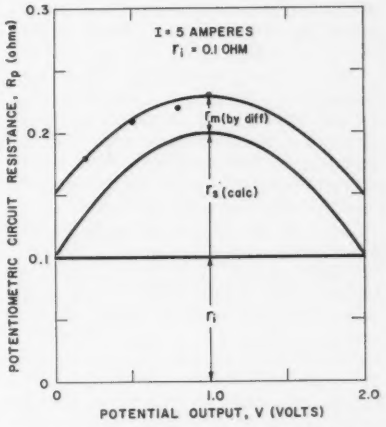

Figure 9—Total potentiometric circuit resistance as a function of potential output.

Concentration	Electrolyte Resistance, r_e at 18 C *
$1\overline{N}$ H_2SO_4	0.702 ohm
$5\overline{N}$ H_2SO_4	0.212
$10\overline{N}$ H_2SO_4	0.204

Figure 10 shows the equivalent cathode resistance, r_c, calculated from the slope of an experimentally measured polarization curve of the test cell cathode. The complex variation of r_c with i is a consequence of the Tafel or logarithmic relationship between hydrogen overvoltage and current. At low current output values, r_c is the predominate source of series resistance while at higher currents it decreases to a low value. Mueller[23] has ingeniously utilized this effect to reduce the series resistance of a modified classical potentiostat. By polarizing with an auxiliary anode, he maintained the cell cathode in the low resistance region throughout the entire anodic polarization

* Conductivity data from International Critical Tables[22]

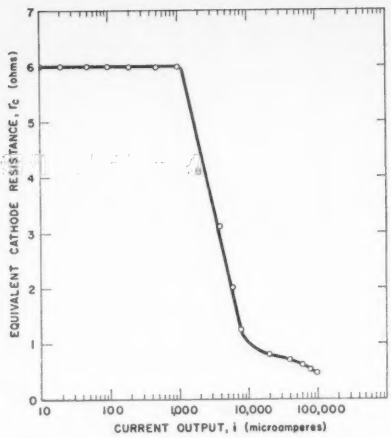

Figure 10—Equivalent cathode resistance as a function of output current.

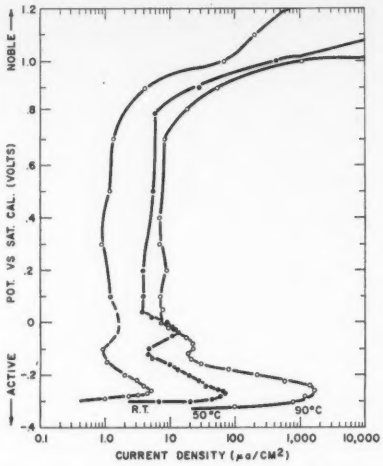

Figure 11—Anodic polarization of Type 304 stainless steel in hydrogen-saturated 1N sulfuric acid.

curve. The apparatus described above may be readily modified to accommodate this technique. However, due to its very low total series resistance, this is unnecessary for the study of many active-passive systems.[24]

The device pictured in Figures 6 through 8 can maintain a potential control of ±1 millivolt over the entire anodic polarization curve for most systems. Potential settings usually remain nearly constant for periods of 10 minutes or more without manual adjustment.

Figure 11 is a series of potentiostatic polarization curves obtained with Type 304 stainless steel in hydrogen saturated sulfuric acid.[(5)] These are "quasi-steady state" curves; that is, at each potential setting, current was measured after a maximum time interval of five minutes. Temperature was controlled within ±1.0 C. The minimum resistance value of the active to passive transition curve for the experiment at 90 C was approximately 28 ohms. The total potentiostatic resistance, R, in this region of the curve was calculated to be less than 11 ohms (I=5 amperes, $r_i = 4.0$ ohms) which is consistent with the fact that the entire anodic polarization curve was measurable and no "potential jumps" were observed. Anodic polarization curves similar to those of Figure 11 have been reported by Edeleanu[1] and Kolotyrkin[6] for 18 Cr - 8 Ni stainless steels in sulfuric acid solutions.

[(5)] These data should not be taken as typical of the behavior of Type 304 stainless steel. A detailed discussion of the passive characteristics of stainless steels will be presented in a future publication.

Summary

1. A quantitative discussion of the classical potentiostat shows that the total series resistance and the maximum current output completely characterize the performance of this instrument.

2. The performance of a classical potentiostat can be increased by reducing its total series resistance and maximum current output. Both quantities can be experimentally determined.

3. A versatile, classical potentiostatic apparatus with an ultimate potential control of ±1 millivolt designed specifically for electrochemical studies of passivity, is described.

Acknowledgments

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List of Symbols

E = Battery potential
E′ = Equivalent series potential
I = Current through inner loop of potential divider network
i = Current output
R = Total series resistance of potentiostatic circuit
R_1, R_2 = Resistances of potential divider legs
R_p = Total series resistance of potentiometric circuit
r = Polarization cell resistance
r_a = Equivalent anode resistance
r_c = Equivalent cathode resistance
r_e = Electrolyte resistance
r_i = Ammeter resistance
r_m = Miscellaneous resistance
r_s = Equivalent series resistance of potential divider network
V = Potential output

Any discussion of this article not published above will appear in the December, 1959 issue

Mechanism of Stress Corrosion Of Austenitic Stainless Steels In Chloride Waters*

By R. W. STAEHLE, F. H. BECK and M. G. FONTANA

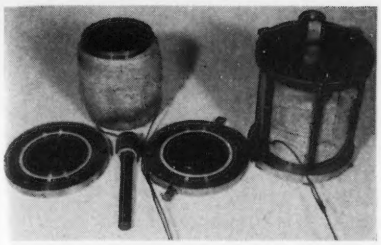

Figure 1—Autoclaves used in stress corrosion cracking studies.

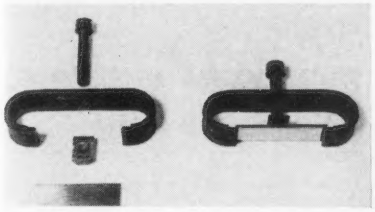

Figure 2—Exploded and assembled views of stress-corrosion specimen and stressing jig.

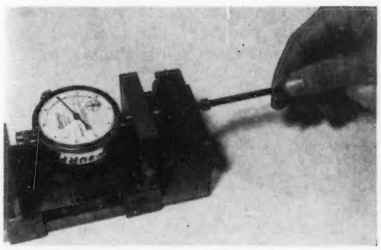

Figure 3—Specimen being stressed in jig.

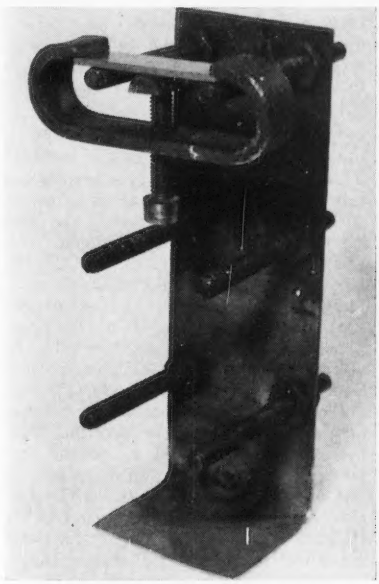

Figure 4—Specimen support rack.

Abstract

Stress-corrosion cracking of austenitic stainless steels was studied under various conditions of stress, chloride concentration, complete immersion of specimens, intermittent wetting and drying, and presence of oxygen. Stress-corrosion cracking will occur at stresses as low as 2,000 psi at 50 ppm NaCl. A three-dimensional analysis of stress-corrosion cracks was made and a mechanism of cracking proposed. 3.2.2

Introduction

THE SUBJECT of stress-corrosion cracking in austenitic stainless steels has aroused much interest because of the extensive application of the alloy, the low concentrations of the chloride ion at which stress-corrosion cracking occurs, and the low stresses at which cracking propagates in the presence of the chloride ion.

The effect of various chloride salts on the stress-corrosion cracking of austenitic stainless steels has been studied extensively by Edeleanu.[1] He shows in general that it is the chloride ion itself that is the culprit, the effect of the cation being relegated to affecting only the conditions of relative time and temperature for cracking.

It is generally agreed that the presence of oxygen is necessary for chloride stress-corrosion cracking in austenitic stainless steels. Eckel and Williams[2] have proposed a chloride-oxygen relationship in the specific conditions of intermittent wetting and drying and alkaline-phosphate treated chloride waters. As the oxygen increases, less chloride is required for cracking. Conversely, as the chloride increases, less oxygen is required. For example, at 220 ppm of oxygen no cracking occurs with 0.1 ppm chloride but does occur at 1.0 ppm. On the other hand, at about 800 ppm of chloride, cracking occurs unequivocally at 10 ppm oxygen, whereas none occurs below 0.1 ppm.

The actual role of the chloride ion in stress corrosion cracking does not seem to be well understood. Numerous studies have shown its relationship to pitting.[2,3,4,5] It appears that the chloride ion is adsorbed on ferrous surfaces[6] in preference to oxygen, and through the mechanism of continuous film breakdown and repair thus forms local anodic areas which lead to pitting.

The lowest stress at which stress-corrosion cracking of austenitic stainless steels will crack in the presence of chlorides apparently is affected by the anion, temperature and the physical nature of the medium which, in turn, control the chloride to oxygen ratio present at the cracking site. Some authors indicate that the minimum stresses required for cracking are on the order of yield,[7,8] while other investigators indicate that stresses as low as 3,000 to 10,000 psi[9,2] will produce cracking. Threshold stresses for cracking have been studied in detail by Hoar and Hines.[10,11] These authors indicate that in boiling $MgCl_2$ the threshold stress for cracking in austenitic stainless steel is about 20,000 psi.

The subject of stress corrosion in all of the prominent alloy systems as well as that in austenitic stainless steels has been studied extensively by many investigators and such an extensive survey of these systems is omitted because of space considerations. The purpose of this paper is to discuss the effect of the relative availability of chloride and oxygen and the applied stress on the morphology of stress-corrosion cracks in austenitic stainless steels.

Procedure and Apparatus

Stress-corrosion cracking tests were conducted in autoclaves constructed of Type 304L stainless steel, as illustrated in Figure 1. These units have a 550 ml capacity and are heated by electric resistance tapes. Several layers of asbestos wrapping are placed on top of the heating elements to provide insulation. Each unit contains a thermocouple well; the temperature is controlled by the use of thermocouples in conjunction with standard type temperature controllers. Fluorocarbon gaskets are used to make all closures and have been satisfactory up to 500 F at a pressure of 680 psi.

The autoclaves are carefully washed before each test run to remove residual chlorides. In starting tests, the autoclaves are assembled around the specimens and specimen supports. Opposite tie bolts are tightened simultaneously to ensure even pressure on the gaskets. Test solutions are placed in the units by means of a funnel passing through the opening provided for the thermocouple well.

Figure 2 shows a specimen and specimen clamp used to apply a stress to the specimen. All parts of the specimen clamp are Type 304 stainless steel. Applied stress at the center of the specimen is determined by deflection as read from the dial gauge (calibrated in 0.0001 inch

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units) shown in Figure 3 according to the following relationship for a beam loaded at the center:

$$S = 12\frac{Ecy}{l^2}$$

where:

S—fiber tensile stress of the specimen at the half length

E—Young's modulus for stainless steel, 29×10^6

c—½ thickness of specimen

y—deflection at center of specimen

l—gauge length over which the deflection is measured

When the deflection corresponding to a selected stress is determined, the specimen is placed in the stressing jig and held against the gauge marks. Figure 3 shows

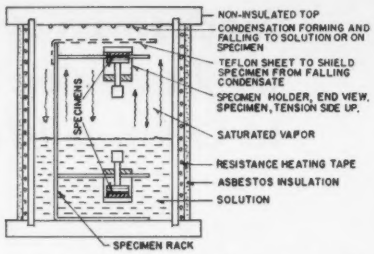


Figure 5—Cross section of autoclave in operation. Specimens are shown in the water phase, vapor condensation (without shield) and saturated vapor (with shield) phase.

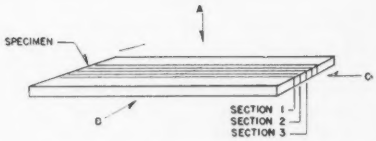


Figure 6—Schematic of sectioned specimen. The surface of the crack is viewed in Direction "A" and the cross section in Direction "B." A series of photographs from Direction "A" and Direction "B" can describe the crack from Direction "C".

the specimen about to be stressed. To stress the specimen the screw of the specimen clamp is turned the required amount.

After the specimen is stressed, it is placed on a rack as illustrated in Figure 4. Fluorocarbon strips placed on the supports are used to insulate the specimen from any galvanic effect of the rack and the autoclave. After the rack is loaded, the entire unit is placed in an autoclave and is ready for testing. For testing in the water phase, the specimens are suspended, tension side down, on the lower part of the rack. For testing under conditions of intermittent wetting and drying, vapor phase, or vapor condensation, the specimens are suspended, tension side up, at the top of the rack. As many as three specimens can be placed on any one level. The rack shown in Figure 4 enables testing to be conducted in the water and vapor phases simultaneously if the autoclave is only partially filled. When the rack is filled, the entire unit is placed in an autoclave as shown in Figure 5 and is ready for testing.

Because of the nature of the application of load by beam deflection, only cracking in its early stages is observed. After cracking has progressed a short distance, stress relief sets in. The specimens, measuring $3.4 \times 1.0 \times 0.225$ cm, were cut from Type 347 stock having the following composition:

Element	Percent
C	0.07
Cr	17.15
Ni	10.36
Si	0.61
Mn	1.48
P	0.018
S	0.015
Nb	0.97

All specimens were held at 2,000 F for ½-hour and furnace-cooled. Three dif-

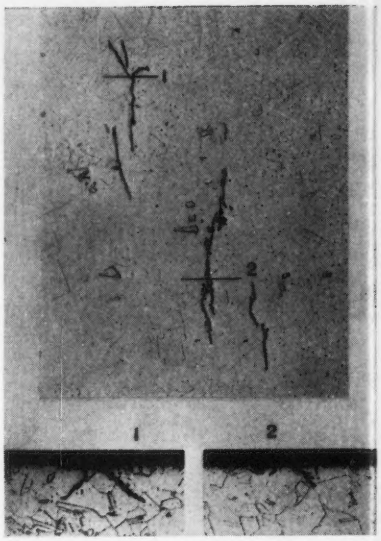


Figure 7—Surface and cross sectional views of cracking on surface A (see Figure 6), abraded with 120 belt with no applied stress. Vapor condensation conditions were present over solution containing 50 ppm chloride. 175X.

ferent surfaces were studied: (1) belt-abraded with 120 grit, (2) pickled six hours in concentrated HCl, and (3) electrolytically polished. The first had residual surface stresses, while the last two did not.

The NaCl concentration of the solution was varied from 50 to 80,000 ppm. In all cases the autoclave was filled with 200 ml of solution. For specific studies on the effect of various gases on the propensity to stress-corrosion cracking, 100 percent oxygen, hydrogen or nitrogen atmospheres filled the remaining 350 ml. Where these gases are not specifically mentioned, air was the gas in the autoclave.

Tests were run for various lengths of time. Since the cracking for all concentrations and for the various physical conditions appeared to be fully developed after four hours at 400 F, the length of the various tests will not be discussed in detail.

Figure 5 shows that the sides, but not the top, of the autoclaves are insulated. This enables a type of intermittent wetting and drying conditions to be present without manually inverting the autoclaves. In the initial experimental work, this type of intermittent wetting and drying was confused with a saturated vapor phase. Cracking was observed when a specimen was suspended in the vapor phase with the tension side up. However, when a fluorocarbon shield was placed above the specimens, no cracking occurred unless the autoclaves were manually and periodically inverted. Since the cracking appeared to be the same whether the condensation from the top fell on the specimen or when the autoclaves were manually inverted, it was decided to use the vapor condensation condition for producing cracks. Such a technique undoubtedly produces an uncertainty in the chloride composition

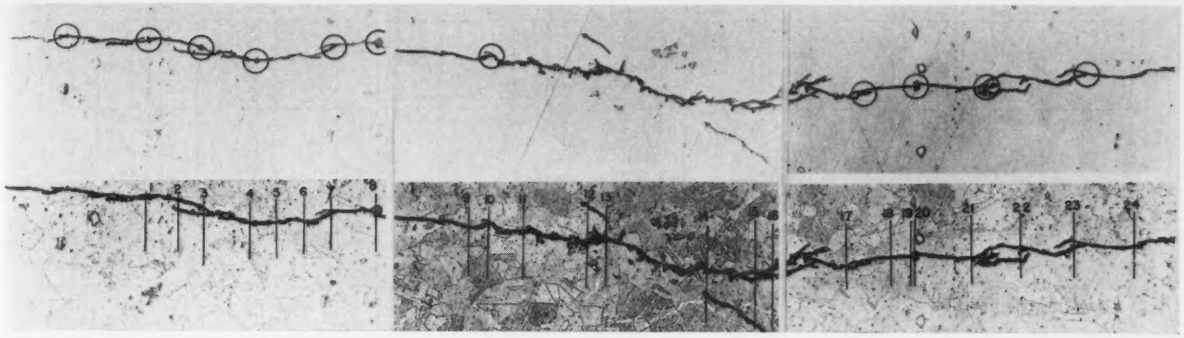


Figure 8—Surface of typical crack, pickled surface, 400 F, chloride containing waters, vapor condensation conditions. Crack is shown etched and unetched. Middle third of crack shows numerous transition points indicating incipient stages of cracking. Remainder of crack shows the transition points more separated. These are encircled and may be either pits or slight direction changes. Numbers refer to cross sections shown in Figure 9.

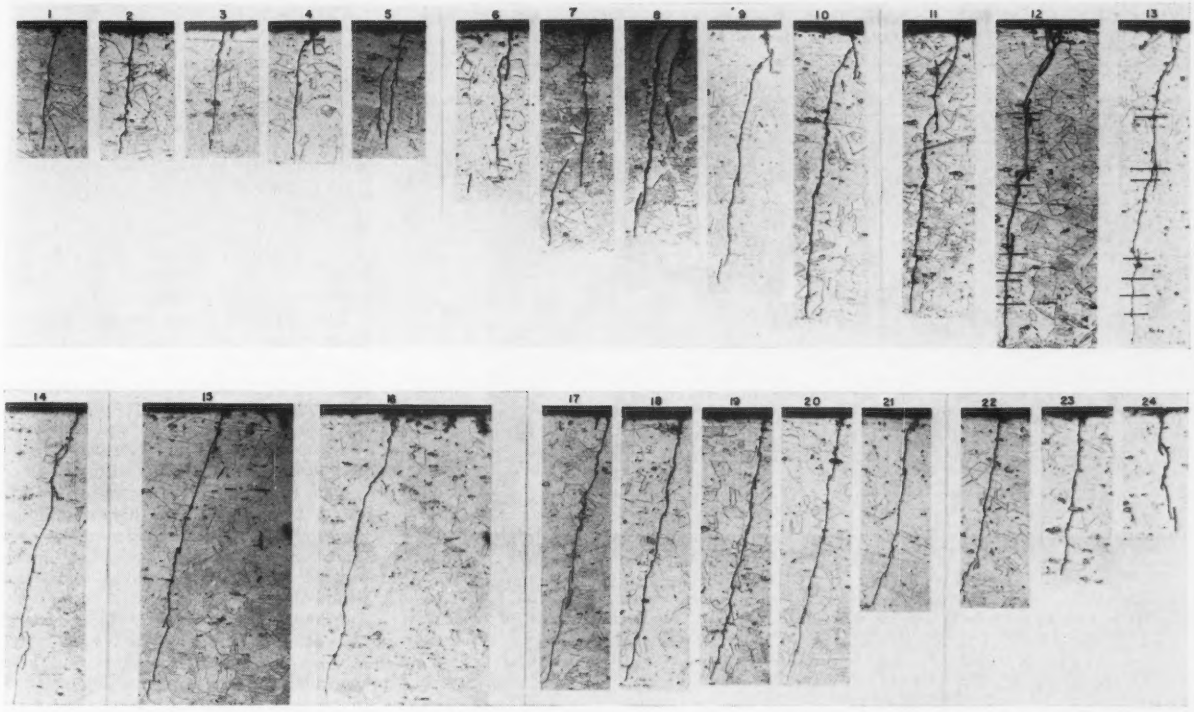


Figure 9—Cross sections corresponding to numbers in Figure 8. Definite changes in direction of crack are apparent and are continuous in several adjacent cross sections. A series of these is indicated on several cracks. 280X.

actually present on the specimen, but the uncertainty is probably no greater than that involved in the manual inversion of the autoclaves.

Surfaces were prepared for photomicrographs by abrading with 240-, 500-, and 600-grit wet-back paper and subsequently by polishing with No. 2 and No. 3 alumina. An electrolytic etch in oxalic acid was used to define grain boundaries, inclusions and cracks.

In cases where it was desirable to show a crack in three dimensions, a special technique was used. The specimen was first mounted in thermosetting plastic, polished and etched. The crack was first photographed on the tension surface of the specimen. The specimen was then polished at right angles to the tension surface to show the depth and character of the crack. Sections of the crack were polished at incremental distances along the crack as shown in Figure 6. In some cases, sections 0.001 inch thick were removed at a time. To give a three-dimensional effect, the depth of cracking was plotted as a function of the surface length of the crack.

Results

Vapor Condensation Condition

The experimental data from the vapor condensation conditions are summarized in Table 1. The solution concentrations given in this table indicate only the initial composition of the solution. Chemical analyses for chloride following a test showed that the solution composition was unchanged during the test. The composition of any solution actually on the specimen is not known, although the chloride on the surface of the specimen probably becomes more concentrated than that of the solution. The results shown in Table 1 indicate that chlorides must be present for the stress-corrosion cracking to occur and that the cracking will occur at as low a concentration as 50 ppm NaCl in the solution and at stresses as low as 2,000 psi on pickled specimens free from residual stresses due to abrading. At low chloride levels, the applied stress did not influence the cracking (i.e., at 50 ppm the specimens stressed at 40,000 psi showed no more tendency to crack than those stressed at 2,000 psi).

The presence of 100 percent oxygen at

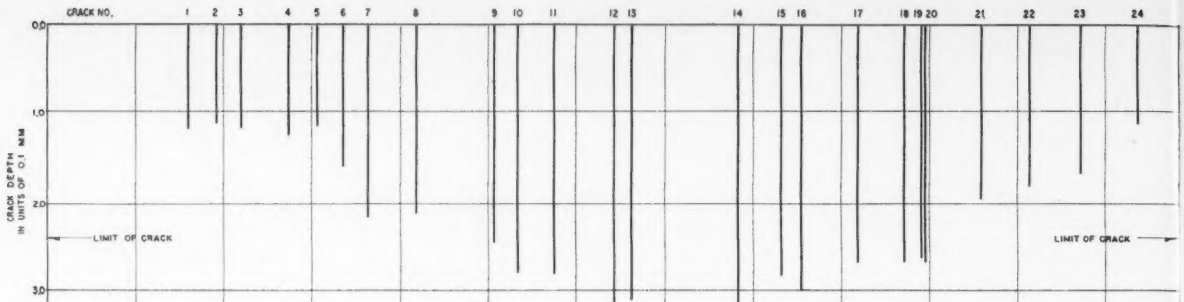

CRACK NO.
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24
0.0
1.0
2.0
3.0
CRACK DEPTH IN UNITS OF 0.1 MM
LIMIT OF CRACK
LIMIT OF CRACK
0.0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0
SURFACE LENGTH OF CRACK (IN UNITS OF 0.1 MM)

Figure 10—Combination of Figures 8 and 9 showing third dimension of crack.

TABLE 1—Results of Tests Conducted in the Presence of Vapor Condensation Conditions in Autoclaves Heated to 400 F

NaCl (ppm)[a]	Applied Stress (1000 psi)	Surface[b]	Cracking
Distilled Water	5, 15, 40	A	No
50	2, 5, 10	P	Susc[c]
50 and 0_2	5, 10	P	Yes
100	5, 15, 40	A	Yes
100	5, 10	P	Susc
100	5, 10	E	No
100 and 0_2	5, 10	P	Yes
150	5, 10	P	Susc
150 and 0_2	5, 10	P	Yes
200	5, 10	E	Susc
875	0[d], 2, 5, 15, 40	A	Yes
875	1, 2, 5, 15, 40	P	Yes
875	3, 10	E	Susc
875 and 0_2	5, 15, 40	A	Yes
875 and N_2	5, 15, 40	A	No
875 and H_2	5, 15, 30	P	No
5,000	5, 15, 40	A	Yes
10,000	0, 2, 4	A	Yes
10,000	5, 15, 40	P	Yes
20,000	3, 5, 15, 40	A	Yes
20,000	3, 5, 15, 40	P	Yes
40,000	5, 15, 40	A	Yes
80,000	5, 15, 40	A	Yes

[a] Where a specific gas is designated after the chloride concentration the remaining 350 ml of the autoclave is filled with the particular gas. Otherwise the gas atmosphere is air.
[b] A-abraded, P-pickled, E-electropolished.
[c] Cracking occurred in about 50 percent of the specimens with no preference for stress level.
[d] The residual stresses due to abrading the specimen were sufficient to cause surface cracking in the absence of applied stress.

TABLE 2—Results of Water Phase Tests Conducted in Autoclaves Heated to 400 F

NaCl (ppm)	Applied Stress (1000 psi)	Surface[a]	Cracking
0	5, 15, 40	P	None
100	5, 15, 40	P	None
875	5, 15, 40	A	None
15,000	20, 35	P	None
		E	None
20,000	20	P	None
		E	None
20,000	40	A	Yes
20,000	30	P	Yes
20,000	35	P	Yes
		E	Yes
25,000	35	P	Yes
		E	Yes
25,000	20	P	None
		E	None
40,000	20, 40	P	None
75,000	20, 35	P	None
80,000	20, 40	P	None

[a] P-pickled surface; A-abraded surface; E-electropolished surface.

50 ppm definitely increases the propensity to cracking. Whereas only half of the specimens cracked at 50 ppm in the presence of air, all of them cracked when the atmosphere was 100 percent oxygen. It also appears that specimens with abraded or pickled surfaces were equally susceptible to cracking at comparable chloride levels, while the electropolished specimens did not crack until 200 ppm NaCl was reached. These results indicate that a smooth surface may present a more difficult environment upon which to nucleate a pit or crack.

The presence of oxygen was shown to be necessary for cracking to occur. Comparing the results at 875 ppm in Table 1, the complete exclusion of oxygen by either nitrogen or hydrogen stops cracking, while the presence of 100 percent oxygen as described above increases the propensity to cracking.

The residual surface stresses on the abraded specimens produced cracking patterns much different from those on the stress-free pickled specimens. On the pickled surfaces, the cracks were perpendicular to the lengthwise axis of the specimen and occurred at the region of highest applied stress (i.e., at the half length). The cracking on the abraded specimens was irregular with respect to the region of highest stress. Cracks occurred as frequently near the end of the specimens as near the center or region of highest stress. Such a crack distribution probably results from disarrayed surface stresses induced by abrading. In some cases, as shown in Table 1, cracking on the abraded surfaces occurred in the absence of applied stress. Several of these cracks are shown in Figure 7. An entire three-dimensional description of a crack from a pickled specimen is shown in Figures 8, 9, and 10. The surface of the crack on the tension side of the specimen is shown in Figure 8, the penetration in Figure 9, and the plot of the penetrations or the third dimension in Figure 10. This crack is typical of those occurring at 2,000 psi.

A cracking pattern of a crack in an abraded surface is shown in Figure 11. This crack occurred at an applied stress of 3,000 psi. Two factors distinguish the cracking on the abraded from the pickled

specimens. First, the cracking on the abraded specimens was only half as deep as that on pickled specimens. Secondly, there is usually a number of smaller cracks in the vicinity of the larger crack, probably resulting from a complex interaction of residual stresses.

In addition to the characteristic cracking patterns described in Figures 8, 9, 10, and 11, some rather unusual patterns occurred on the pickled specimens. Figure 12 shows what appears to be a "circular" crack. The direction of subsurface cracking is not perpendicular to the tension surface, probably because of the complex stresses produced by the initial cracking pattern.

Throughout the photomicrographs there are a large number of pearl-like inclusions, the exact composition of which is not known but is probably a nitride. These inclusions have been observed to arrest the progress of a crack as shown in Figure 13A and have no retarding effect on crack movement as shown in Figures 13B and 13C. Figure 13B shows that the inclusion has actually been bypassed by the crack, whereas the inclusion in Figure 13C exhibits an apparent brittle fracture.

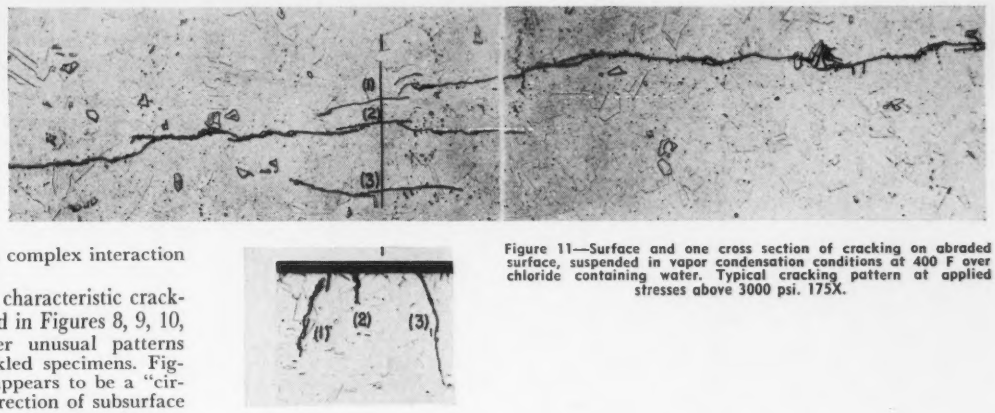


Figure 11—Surface and one cross section of cracking on abraded surface, suspended in vapor condensation conditions at 400 F over chloride containing water. Typical cracking pattern at applied stresses above 3000 psi. 175X.

Water Phase

Table 2 summarizes the results of the water-phase testing. These results present a much different picture of stresses and chloride concentrations. Instead of cracking at the low concentrations, no cracking is observed until 20,000 ppm is reached, and no cracking is observed at concentrations greater than 25,000 ppm. The stresses required for cracking, 30,000 psi or above, are much higher than the mere 2,000 psi required in the vapor condensation conditions.

Although the susceptibility to cracking does not change with surface preparation, the nature of the cracking changes markedly. The cracking of the pickled surfaces is shown in Figures 14 and 15; the cracking of the abraded surfaces is shown in Figures 16 and 17. Note the single crack when there are no complex surface stresses. The character of the cracks as they progress through the metal should be compared with the cracks occurring in the vapor condensation condition. It appears that there was much more electrochemical attack associated with the cracking in the water phase.

Crack Propagation

A detailed study of the crack propagation kinetics was not possible with the equipment employed in this study; however, a cursory study was made for purposes of comparison with available data. By holding the autoclaves at 400 F for 0.5, 1.0, 2.0, 2.5, 3.0, and 6.0 hours and using stress levels of 3,000 and 7,000 psi, it was determined that cracks begin propagating in about 0.5 hour and are fully developed between 2.0 and 6.0 hours. Since the maximum crack depth

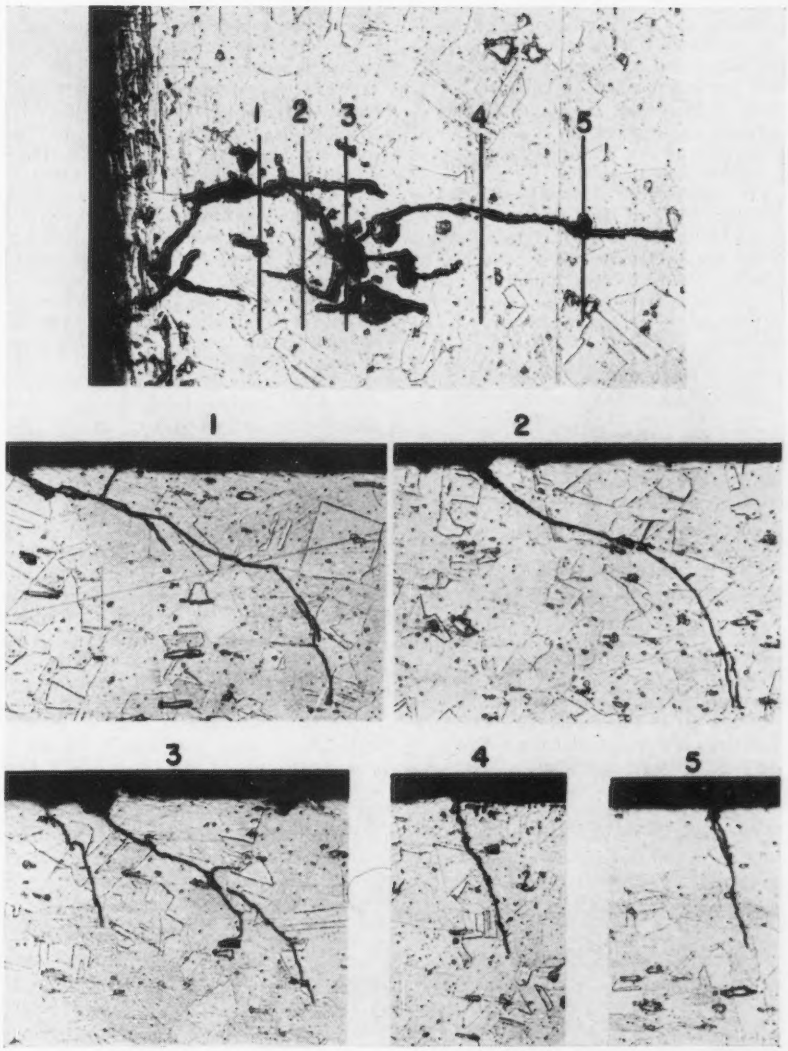
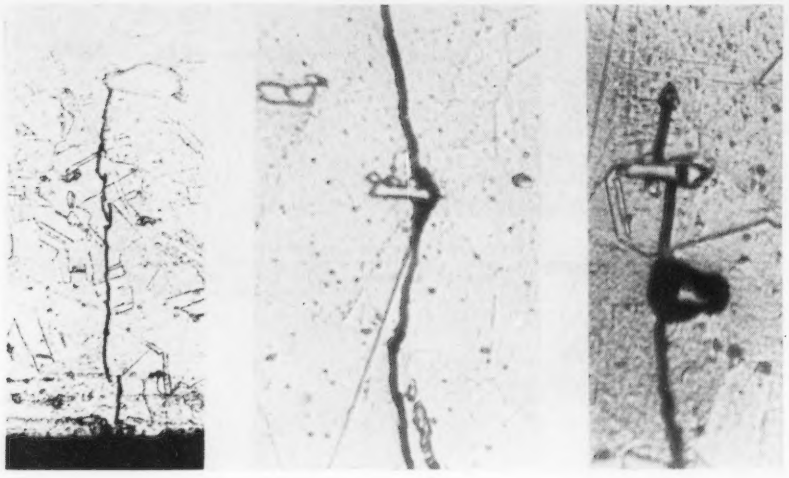


Figure 12—Surface and associated cross sections of cracking on pickled surface. Solution 875 ppm NaCl, 2000 psi, held at 400 F for three days, vapor condensation conditions. Cross sectional views show the crack proceeding non-perpendicular to the direction of applied stress. 310X.



A B C

Figure 13—Inclusions in the crack path. Pickled surface 2000 psi. Vapor condensation. Photomicrographs are, left to right: crack arrested, 350X; crack by-passed, 1370X; brittle fracture in inclusion, 1370X.

observed was 0.4 mm, the rate of cracking probably lies in the range from 0.2 mm/hr to 0.067 mm/hr. These figures are in approximate agreement with those of Hoar and Hines,[10] who suggest that cracks in austenitic stainless steels propagate at the rate of 1-4 mm/hr in a boiling $MgCl_2$ solution under stresses on the order of yield. Since actual brittle fractures occur at much higher velocities, it might be concluded that the progress of the stress corrosion cracks is dependent upon an electrochemical action for movement, while the stress plays the role of activating or helping to initiate the cracking.

Discussion of Results

The annealed ultimate tensile strength of the 300 series of stainless steels is approximately 85,000 psi; the yield strength is approximately 35,000 psi at 0.2 percent offset; and the threshold stress for stress-corrosion cracking in the vapor condensation condition may be lower than 2,000 psi. The burden of this discussion is to suggest explanations for such insidious behavior.

Crack Initiation

The chloride concentration and stresses at which cracking occurs in the water phase and vapor condensation conditions give a clue to the mechanism of crack initiation. It appears that the mechanism of crack initiation is related to the available oxygen, specific concentrations of the chloride ion, and the stress level. The significance of these factors lies in the comparison between the conditions leading to cracking in the water phase and vapor condensation conditions. In the vapor condensation condition there is a ready availability of oxygen, but the chloride level in the water below the specimen was much lower than that required for cracking in the water phase. It is suspected that the chloride achieved high local concentrations through a constant cycle of drops of solution falling on the specimens and the subsequent evaporation. This high local chloride concentration together with the abundant oxygen in the air produced a type of pitting or initial attack that lead to the cracking at the unusually low stresses.

In contrast to the cracking at the low chloride concentrations in the vapor phase, there is no cracking at the low chloride concentration of the water phase. Cracking in the water phase did not proceed until there was sufficient chloride concentration to produce cracking with the relatively low oxygen concentration in the water. The disparity in the threshold stresses between the vapor condensation and the water phase is probably accounted for by the type of pit formed under the conditions of chloride and oxygen concentrations present. Whereas the pit formed in the vapor condensation conditions would have a high depth-to-radius ratio, the same ratio for the pitting in the water phase would be much lower and, hence, would require a higher applied stress to propagate. There is also no obvious reason for the resultant stresses to be extremely high because the cracking does not necessarily propagate by mechanical fracture. It can just as easily propagate by a predominantly electrochemical mechanism with the applied stress supplying just enough strain energy or plastic deformation to exceed the energy requirements for crack propagation. The variance in the types of pitting in the two conditions can be seen in Figures 8 and 16. The pits on the pickled specimens in the vapor condensation conditions have a small surface diameter (although no data are available on the actual pit configuration just prior to crack initiation), and the pits in the water phase have a much larger surface diameter, in some cases by a factor of about 20. These observations tend to confirm the above hypothesis.

This explanation for the apparently dissimilar conditions that lead to cracking would not seem to account for the absence of cracking at the higher chloride concentrations in the water phase. The only possible explanation would be that, with the higher chloride levels, there is a decrease in the solubility of oxygen, which has been reported by Asselin and Rohrman[12] for another temperature range.

A verification of the saucer-like depressions occurring in specimens immersed in solution has been made by Hoar and Hines,[10] who have observed that, on wire specimens in the aqueous $MgCl_2$, the pitting actually takes the form of saucer-like depressions.

There is no obvious answer to the question of how the sites for crack initiation are chosen. The most likely mechanism involves the continuous breakdown and repair of the surface film. The film breaks down and the chloride ions adsorb on the surface before the oxygen and lead to the formation of an anodic area. This tendency for the chloride ion to displace the oxygen has been verified by Hackerman and Stephens.[6]

Considering next the macro scale, the cracking on the stress-free pickled surfaces always occurs at the half length. This happens in spite of the fact that, when a stress at that point might be 15,000 psi, the stress near the end might be 1,000 to 2,000 psi which, on other specimens stressed to this level at the center, crack at the center. In other words, the cracking always occurs at the center of the specimen in the region of the highest stress. This is not easily understood on the basis of stress relief of a beam-loaded specimen since the cracking might initiate at any location when the specimens are highly stressed. It is probable that the galvanic effect of a

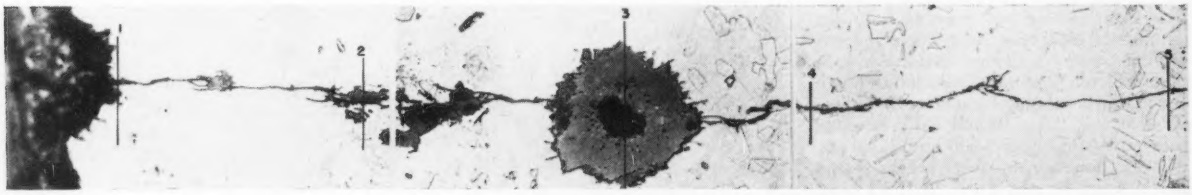


Figure 14—Surface (Direction A) of crack on specimen immersed for five days in 400 F water having 20,000 ppm NaCl, stressed to 40,000 psi, and pickled surface. Numbers refer to corresponding cracks in Figure 15. 280X.

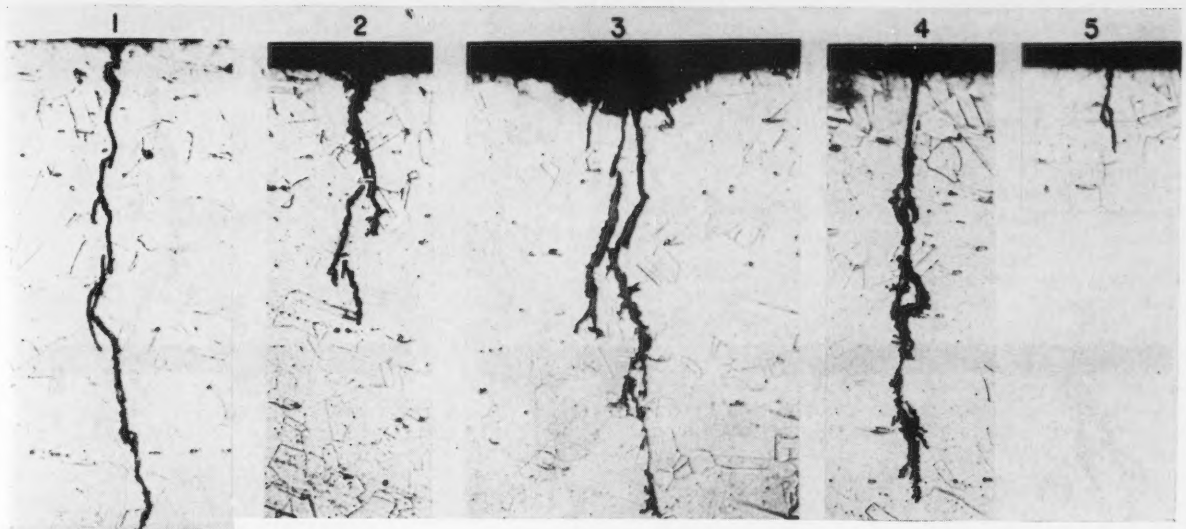


Figure 15—Cross section of cracks (Direction B) shown in Figure 14. 335X.

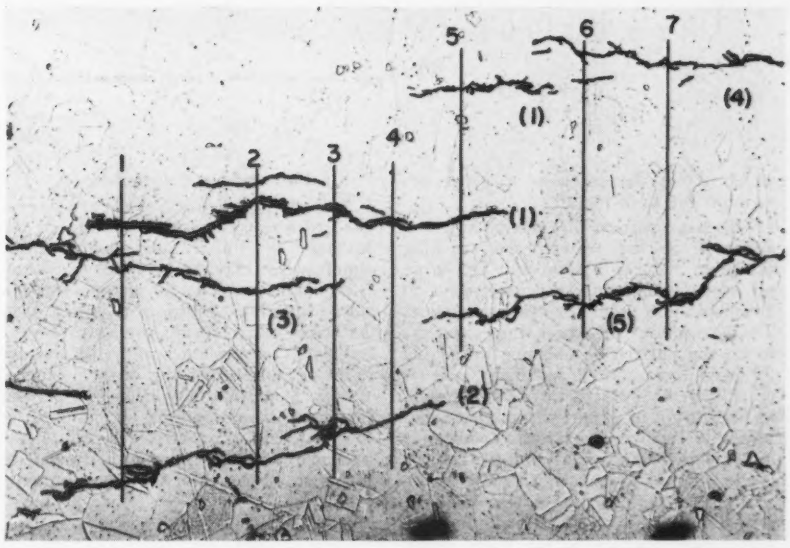


Figure 16—Surface (Direction A of cracks, abraded surface, applied stress 40,000 psi, immersed in water phase of 20,000 ppm NaCl held at 400 F for five days. Numbers in brackets refer to particular cracks; numbers not in brackets refer to successive sections. 250X.

highly stressed region can accentuate the pitting tendency at the center of the specimen.

The cracking patterns of the abraded specimens apparently confuse the above hypotheses. The cracking frequently occurs at any location on the specimen, and the prevalence of pitting is not obvious, although pits were apparent on many of the cracks. Since there is not positive control on the cracking of the abraded specimens, as in the case with the stress-free surfaces, no positive hypothesis can be advanced on the basis of the cracking patterns. The presence of the residual stresses probably obscures the mechanisms which are more readily apparent on the stress free surface.

Crack Propagation

For the purposes of the discussion on crack propagation only, the cracking on the stress-free pickled surfaces will be considered since the cracking patterns on the abraded surfaces have been disrupted by the presence of residual stresses and the interpretation of the cracks would be clouded with extraneous factors.

Figures 8, 9, and 10 will be the basis for the proposed mechanism of stress-corrosion crack propagation. The most significant single fact in the interpretation of crack propagation is that only between 1,000 and 2,000 psi are required for cracks to propagate under conditions of vapor condensation. Cracking at the higher stresses in the water phase seems more credible since the stress required for cracking is only a factor of 3-4 less than ultimate strength rather than a factor of about 50 in the case of the vapor condensation.

On the basis of Figure 8, cracking in the vapor condensation condition may be divided into stages. First is the pitting stage. Note the large pit in the approximate center of the crack. This pit coincides with a relatively large circle of reaction products observed on the specimen when it was removed from the autoclave. The next stage is actually the first stage of crack propagation. The character of the center one-third of the crack is basically different from the rest of the crack. At this point the crack is not sufficiently deep to give an effective stress raiser.

The second stage of crack propagation can be interpreted as a series of continuous burst of crack progress. The crack has achieved a critical depth-to-root radius ratio and there is less difficulty in maintaining a continued crack movement. If the applied load in this experiment were constant, catastrophic cracking would be the next stage of cracking; however, the stress relief inherent in beam-loaded specimens causes the crack to stop before this stage is reached. This periodic mechanism observed on the surface of the crack is obvious in Figure 9,

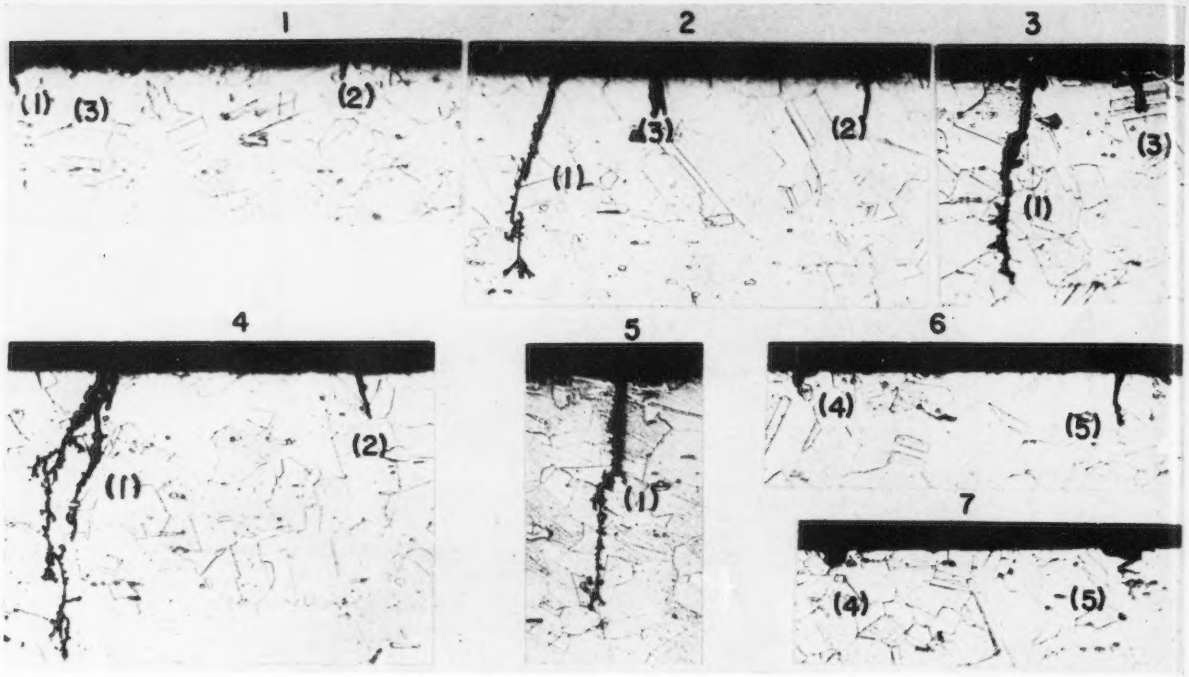


Figure 17—Cross section of cracks shown in Figure 16. 280X.

which shows the crack as it progresses through the bulk metal.

The same pattern of cracking stages is apparent in the water-phase cracking shown in Figures 14 and 15. The large pit or depression in the center seems to have been the point for crack initiation. To the right of the crack there appears to be the pattern of abortive branching followed by a more continuous type of crack. In the bulk metal there is again an indication of a periodic progress, but points of change are not so definite. It appears that the actual progress of the crack is obscured by a much more extensive electrochemical attack than was observed in the vapor condensation conditions. The cracking in the water phase seems to be much more ragged, probably resulting from a more abundant supply of chloride ions in that water phase than in the vapor condensation condition. In fact, the ragged appearance resulting from more extensive electrochemical attack probably can be correlated with the large surface area of the pits.

A tentative conclusion based on the mechanism of crack propagation would be that the cracking is primarily electrochemical, with the stress playing the role of film breaker or, at the most, flowing the metal at the base of the crack which would lead to a high strain energy. On the basis of the low stress and the crack velocity connected with the cracking in the vapor condensation conditions, a type of periodic electrochemical mechanism is much more credible than one in which the stress actually contributes to the crack progress by a fracture mechanism.

Summary and Conclusions

1. Type 347 stainless steels are susceptible to stress corrosion cracking in the presence of 400 F chloride-containing waters when exposed to vapor condensation conditions. The threshold stress applied for this condition is lower than 2,000 psi and chloride concentrations as low as 50 ppm in the water will produce cracking. Increasing the oxygen concentration from that in air to 100 percent increases the probability of cracking at low chloride concentrations, while the presence of 100 percent hydrogen or nitrogen above the solution eliminates cracking.

2. Type 347 is susceptible to stress-corrosion cracking under highly specific conditions when immersed in chloride solutions at 400 F. The solution concentration range of susceptibility is 20,000 to 25,000 ppm NaCl and the stresses must exceed 30,000 psi.

3. Type 347 does not crack in a saturated vapor phase (no condensation) over an 875 ppm NaCl solution at 400 F.

4. Cracking progresses periodically. After the initial pit forms, the crack progresses in short abortive branchings. In the second stage of cracking, the cracks move in longer bursts of 2-10 grains in length. The onset of this stage is probably controlled by a certain stable crack size reached in the first stage. The third stage would be catastrophic cracking if the specimen were under constant load instead of being beam-loaded.

5. On the basis of the cracking at the low stresses, it is proposed that the actual crack movement results from electrochemical action with the stress serving to provide a local region of highly stressed metal at the base of the crack. This highly stressed metal provides a local anodic area for preferential electrochemical dissolution.

Acknowledgment

The support of this investigation by the Office of Naval Research is gratefully acknowledged. This paper is based on a thesis[13] presented in partial fulfillment of the degree, Master of Science, at The Ohio State University.

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DISCUSSION

Question by E. H. Phelps, Applied Research Laboratory, U. S. Steel Corporation, Monroeville, Pennsylvania:

You pointed out that cracking did not occur in the water phase at high chloride concentrations. Do you have any explanation for this effect?

Reply by R. W. Staehle, F. H. Beck and M. G. Fontana

Cracking will occur in the water phase at high chloride concentrations but higher stresses and longer times are required. Cracking under "vapor condensation" conditions is rapid at very low applied stresses.

Questions by Andrew Dravnieks, Standard Oil Company of Indiana, Whiting, Indiana:

1. We found that cracks have blunt ends, supporting the electrochemical rather than the mechanical mechanism of cracking.

2. Could you outline the metallurgical prerequisite for chloridic stress corrosion cracking? We believe that neither Cr nor Ni has to be present in alloy, since Invar as well as Mn-based austenitic steels, or "C-N, no Ni" austenites also crack. Furthermore, low-Ni, theoretically non-austenitic, stainless also cracks.

Reply by R. W. Staehle, F. H. Beck and M. G. Fontana:

1. We agree with Dr. Dravnieks on the electrochemical mechanism.

2. It would be difficult to outline a metallurgical prerequisite for chloride stress corrosion cracking since several alloy systems crack. You may recall our movie on cracking of a magnesium base alloy.

Work completed since the presentation of this paper shows that 18-8 cracks more readily under these test conditions than a straight chromium stainless steel and also more readily than plain carbon steel.

Any discussion of this article not published above will appear in the December, 1959 issue

DISCUSSIONS ON TECHNICAL ARTICLES ARE SOLICITED

Discussions on technical articles published in Corrosion will be accepted for review without invitation. Discussions must be constructive, accompanied by full substantiation of fact in the form of tables, graphs or other representative data and be submitted in three typewritten copies. In cases where illustrations are submitted, at least one copy of figures should be of a quality suitable for reproduction.

Authors will be sent on request a copy of the NACE Outline for the Preparation and Presentation of Papers.

Discussions will be reviewed by the editor of Corrosion and will be sent to the author of the paper discussed for his replies, if any. Publication will be in the Technical Section with full credit to the authors together with replies. Discussions to papers presented at meetings of the association may be submitted in writing at the time of presentation or later by mail to the editorial offices of Corrosion, 1061 M & M Bldg., Houston 2, Texas.

Corrosion of Reinforcing Steel In Concrete in Marine Atmospheres*

By D. A. LEWIS* and W. J. COPENHAGEN**

Abstract

Where a reinforced concrete structure is exposed to a marine atmosphere, serious corrosion of the reinforcing steel sometimes takes place within a relatively short time and the concrete cover round the reinforcing is spalled off.

This phenomenon, examples of which are described, constitutes a major problem in South Africa, particularly in coastal areas. No exhaustive work on this particular subject is described in the literature and because of the nature of concrete, conventional methods of corrosion prevention find little direct application.

The reaction proceeds by electro-chemical mechanisms whose emf's derive from differences in pH, aeration or other factors but whose severity is influenced mainly by presence of sodium chloride in the concrete.

Accelerated corrosion tests in a salt spray cabinet using steel reinforced mortar specimens indicated that factors of considerable significance are cover to the steel, protective coatings to the mortar, and the curing, cement content and water cement ratio of the mortar. The effects of these variables in the corrosion of the steel are discussed.

A comprehensive series of both accelerated and natural exposures of reinforced concrete specimens in progress is outlined.

6.6.5

Introduction

IN THE Union of South Africa it has been observed frequently that reinforced concrete structures exposed to marine atmospheres deteriorate in a relatively short period. Minor sections such as balusters and hand rails of bridges have sometimes disintegrated in four to five years. There is evidence that some of the main beams are also affected.

On the South Coast of Natal the conditions are particularly severe. Halstead[1] has described how a brown stain will appear on the reinforced concrete surface. At this stage there is no evidence of cracking. Later, a fine crack appears coincident to the steel underneath the surface; the crack widens with time and eventually the concrete over the reinforcement is spalled off and the reinforcing steel exposed to the atmosphere.

The deterioration does not always follow this pattern exactly; often the brown stain is absent. However, the result is always the same. By the time the crack appears, the steel has corroded. When the crack has widened to the point where spalling of the concrete is imminent, the probability is that the reinforcing steel has been appreciably reduced in section with the associated loss of structural strength.

From the age of the structure and the original diameter of the reinforcing steel in the case of two South Coast bridges, the figure of 0.006 to 0.010 inches per year was deduced as a corrosion rate.

This figure is considered severe when compared to the atmospheric corrosion rates of 0.007 inches per year for Sheffield, England (a severely polluted atmosphere) and 0.035 inches per year for a coastal tropical location. These figures are quoted for bare steel whereas the reinforcing steel was covered with concrete.

That the problem of corrosion of reinforcing steel has reached major proportions is evidenced by the attention it has received in South African journals[1,2] and the concern of public and other bodies responsible for reinforced concrete work in South Africa. There is also evidence that the problem has been experienced in other countries,[3,4,5,6] but its magnitude is considerable in South Africa.

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Corrosion Processes

Because the disintegration of reinforced concrete is essentially a problem of the corrosion of steel in a specific environment, the chemical nature of concrete as it affects the corrosion of steel will be considered.

Set concrete made from Portland cement, sand, stone and fresh water will contain the following:

1. The sand and stone which may in general be regarded as inert.

2. Particles of un-ground cement clinker, also inert.

3. Calcium hydroxide and small quantities of other alkalis.

4. Hydrated cement minerals (calcium silicates, calcium aluminates and calcium alumino ferrites).

5. Varying amounts of uncombined water.

6. Calcium carbonate and calcium sulphate.

Wherever any moisture is present, concrete can thus be regarded as an electrolyte containing mainly calcium hydroxide. In fact, the only ions present which could conceivably influence reactions with steel under conditions normally prevailing in reinforced concrete structures would be the hydroxyl, calcium, sodium, potassium, carbonate and sulphate ions.

Behavior of Steel in Hydroxides

The existence of a highly protective film of gamma ferric oxide formed on the surface of steel in alkaline solutions has been shown by Mayne and Menter.[7] Freshly abraded iron becomes passive when immersed in decinormal sodium hydroxide containing dissolved oxygen. Weak areas in the air-formed film are first repaired by the formation of ferrous hydroxide which then reacts with oxygen to form mainly cubic Fe_3O_4 and gamma Fe_2O_3. Later repair may occur by direct electrochemical production of gamma ferric oxide. In this way, the film thickens until it becomes impervious to ions and passivity ensues. This system holds in a like manner for steel in solutions of calcium hydroxide.

In the presence of sodium chloride, however, the concentration of hydroxyl ions necessary to stifle attack on the steel rises steadily with the chloride ion concentration. As soon as chloride ions are in excess, the main anodic product of corrosion will be ferrous chloride, and further attack will therefore not be stifled. This effect is illustrated in Figure 1.[5]

If, for some reason, the hydroxyl ion concentration of the solution in contact with the steel should fall, the protective film would be disrupted and corrosion could proceed.

Because gamma Fe_2O_3 film is capable of conducting electrons from the steel to the outer oxide surface, steel surfaces having this film could act as efficient cathodes.

Factors Affecting Passivity of Steel in Concrete

It has been shown that clean steel is rendered passive by concrete, or rather

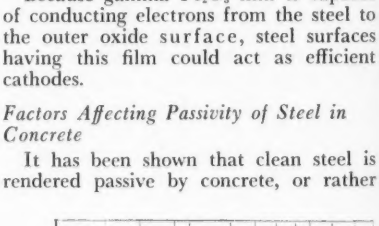

Figure 1—Potential-time curves for steel in cement extracts containing various amounts of salt.

Figure 2—Cracking, spalling and corrosion in a polluted industrial atmosphere in the Transvaal.

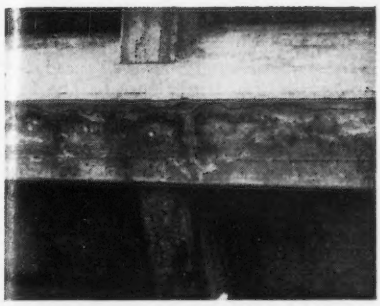

Figure 3—Cracking prior to spalling on underside of a south coast road bridge.

by the action of the hydroxyl ions in concrete in the absence of any aggressive ions. When dealing with reinforced concrete structures in practice, however, there are certain factors that will influence the passivity of steel. If the conditions listed below change, it is possible for the concrete to act as an electrolyte in which corrosion cells will operate.

1. Quality of the concrete: If cracks or honey-combing are present in the concrete, the atmosphere can penetrate to the steel thus exposed, and normal atmospheric corrosion of steel will proceed at these points.

2. Concrete permeability: There is ample evidence that concrete is permeable to varying degrees. The permeability depends on numerous factors[9, 10] such as water-cement ratio of the mix, aggregate size and grading, richness of mix, method of compaction, curing, etc.

Furthermore, the permeability of a given concrete section probably is not a constant quantity but varies with time and history. Because concrete is not an ideally homogeneous material, precise measurement of permeability from the smallest to the largest degrees, and more particularly, the measurement of local differences in permeability in the same structural member, has not yet been satisfactorily achieved.

Since the passivating action of concrete on steel, due to its high pH, can be maintained only by its physical protection and durability, i.e., its ability to exclude inimical ions from near the metal surface, the factor of permeability is of the utmost importance.

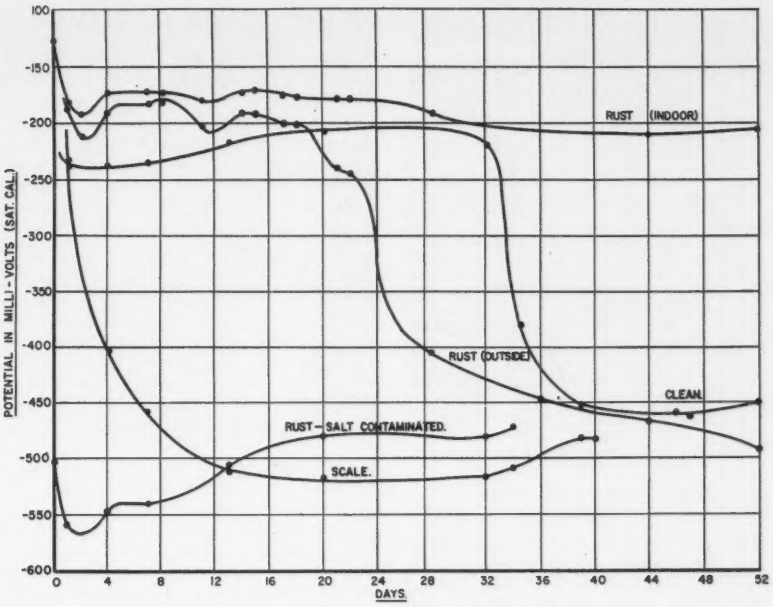

Figure 4—Effect of surface condition of steel.

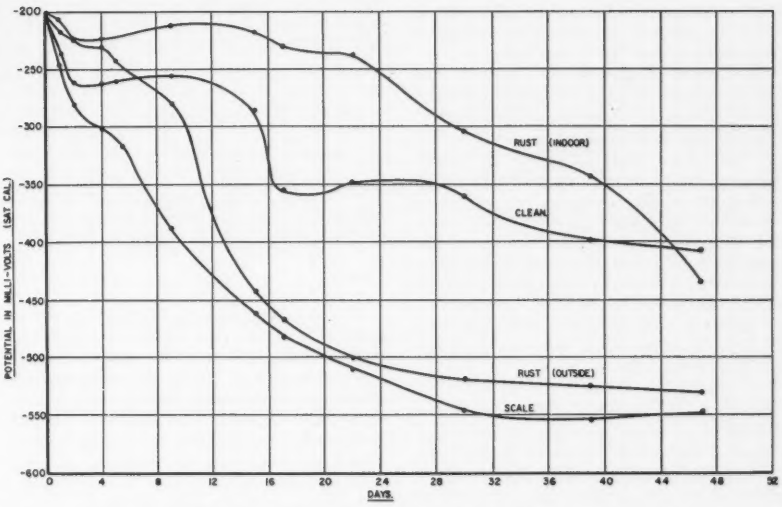

Figure 5—Effect of surface conditions of steel (1 inch cover).

3. Environment of the concrete structure: When embedded in concrete, reinforcing steel in the normal unpolluted Transvaal Highveld atmosphere rarely corrodes. Figure 2 illustrates the effect on reinforced concrete when the atmosphere is contaminated by spray from industrial operations involving brine. Such corrosion, usually not severe, is frequent in coastal locations.

A typical example is shown in Figure 3. The two environments have in common the presence of the chloride ion and a high humidity over and above the usual constituents of the atmosphere. Atmospheric environment in which a reinforced concrete structure is located is thus highly significant.

However, generalizations may be misleading because a structure in a geographical area of aggressive atmosphere can remain unattacked if some feature of the immediately local topography shelters it. Thus, different areas of the same bridge may suffer entirely different corrosion rates due to the sheltering of the one section. This aerodynamic effect is particularly noticed on seaward and landward sides of a structure.

Local humidities may vary considerably, for example over a river estuary, and could be widely different from place to place and from the recorded observations of a nearby meteorological station.

4. Steel surface: Concrete acts as an anodic inhibitor by deposition of a protective film at the anodes. At the same time, by obstructing the diffusion of oxygen to the metal surface, it impedes the normal cathodic reaction for which the

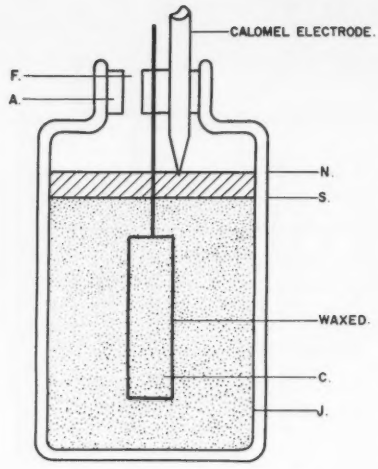

Figure 6—Experimental half cell.

presence of oxygen is necessary. However, the amount of hydroxyl ion required to stifle corrosion increases as chloride ion concentration increases. Thus steel whose surface is contaminated with salt is likely to corrode in concrete sooner than clean steel. Actively rusting steel or steel whose surface retains millscale is likely to have less corrosion resistance than steel which has been cleaned.

In Figures 4 and 5 the effect of the surface condition on the inhibitive period before corrosion commences is shown for exposed and mortar-covered steel.

5. Cover of concrete over the steel: This is a factor which must be considered with permeability because two inches of permeable concrete will yield less protection to steel than a quarter-inch of impermeable concrete. However, when cover varies from point to point along a length of reinforcing steel where the concrete is of uniform quality, the result would be the same as if the cover were constant but the quality varied.

Corrosion Cell Action

Bird[8] has described how salt, windborne or deposited on the concrete surfaces by sea spray, can dissolve in surface moisture and enter the pores of concrete by capillarity or during sudden wetting after a dry period. After evaporation, salt deposits will remain in the pores of the concrete. If more salt penetrates than is leached out, it is possible for significant chloride concentrations to develop within the concrete.

Such penetration and accumulation of salt will occur preferentially with oxygen, carbon dioxide and moisture in those areas of the concrete, large or small, which are more permeable than other areas. For a given thickness of cover, the permeability of the concrete will determine the penetration rate for a given environment.

At areas of highest permeability, penetration will occur relatively quickly down to the steel surface, and the following effects will be produced:

1. Oxygen penetration will depolarize the cathodes of a corrosion cell.

2. Carbon dioxide penetration will carbonate the free lime in concrete thus reducing the pH locally. (If only the surface of a lime particle is carbonated, such a particle may possibly be discounted as contributing to a high pH.)

3. Sodium chloride penetration will reduce the inhibitive effect of the hydroxides and also lead to a considerable reduction in electrical resistivity of the concrete.

4. Moisture penetration will influence penetration of all the above materials. In the absence of moisture all corrosive factors will become inoperative.

Another overall effect of the ingress of sea water will be the production of small pockets, or micro-climates, within the concrete pores where hygroscopic sea salts tend to retain moisture. Thus, while externally the humidity may be temporarily low, the humidity of the microclimate may exceed the critical humidity allowing rapid corrosion to proceed.

With the penetration of moisture containing oxygen, carbon dioxide and sodium chloride, the steel will no longer be passive and micro-cell corrosion (where the anodes and the cathodes are microscopic distances apart) will proceed where the pH is low. Since the micro-environment is no longer inhibitive, innumerable micro-cells may operate, but the most common would be the millscale cell and differential aeration cell as follows:

Steel (Anode)	Aerated solution	Millscale (Cathode)

Steel (Anode)	Low oxygen concentration (Chloride solution)	High oxygen concentration (Chloride solution)	Steel (Cathode)

Micro-cell corrosion of this nature is similar to normal atmospheric corrosion. But in addition to these micro-cells it can be seen that, where the permeability of the concrete varies from place to place on a macro-scale, it is possible for various differential macro-cells to operate. The variables which will influence the macro-cell operation are amount of water present, amount of chlorides present, pH of the moisture at the steel surface and amount of oxygen available.

The macro-cells which could conceivably operate, are detailed individually in Table 1. In practice some or all would be operating simultaneously and only the one with the dominant emf would be important.

Experimental Investigation of Various Macro-Cells

Electrochemically, current flowing between two members of a cell is proportional to the difference in single electrode potential (or solution potential) of the two half cells. The cell will function according to the laws of Ohm and Faraday.

This solution potential is the compromise value between the potential of the local anodes and that of the local cathodes on the surface of the steel. Normally the corrosion of an immersed steel surface is dependent on the rate at which oxygen arrives at the cathode (cathodic control), and the potential of the local cathode is polarized down to the potential of the local anode. The single electrode or measured potential of the steel therefore normally approximates the open circuit potential of the local anodes.

The experimental half cell used by Bird[8] is illustrated in Figure 6. The flat mild steel specimen C (3 in. × ½ in.) degreased, pickled, dried and waxed round the edges and stem, was placed in the stoppered jar. Salt-free dry sand was poured in to the level S. The appropriate solution was then pippetted in until a slight excess was observed above the level of the sand. The saturated calomel reference electrode was employed for single electrode potential measurements.

Cell No. 1 (described below) corresponds to steel embedded in a section of concrete both permeable and impermeable in parts, where local concentrations of sodium chloride could build up.

Cell No. 1

Steel	Neutral salt solution of low concentration	Neutral salt solution of high concentration	Steel

The following theoretical considerations should be noted:

1. The well-known Nernst equation

$$E = E_o + \frac{RT}{NF} \log_e C.$$

holds for the potential of a metal with respect to its own ions in solution only.

2. In the concentration type cell, the half cell with the lower metal ion concentration becomes the anode.

TABLE 1—Possible Macro-cells in Corroding Reinforced Concrete

Cell No.	Relatively Permeable Concrete		Relatively Impermeable Concrete	
1	Steel (Anode)	Neutral salt solution (Conc. a)	Neutral salt solution (Conc. b < a)	Steel (Cathode)
2	Steel (Anode)	Carbonated cement	Uncarbonated cement	Steel (Cathode)
3	Steel (Anode)	Carbonated cement and salt	Alkaline cement	Steel (Cathode)
4	Steel (Anode)	pH = X salinity = y	pH = a > X salinity = b<y	Steel (Cathode)
5	Steel (Cathode)	High oxygen availability	Low oxygen availability	Steel (Anode)

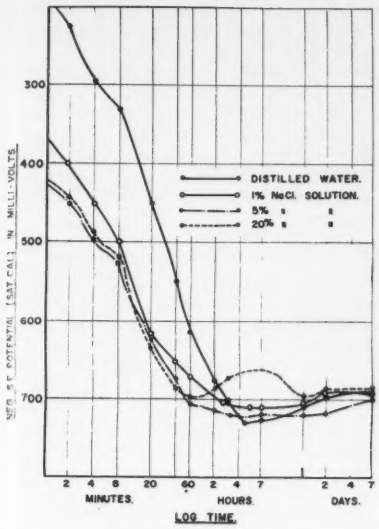

Figure 7—Time potential curves for steel in various neutral salt solutions.

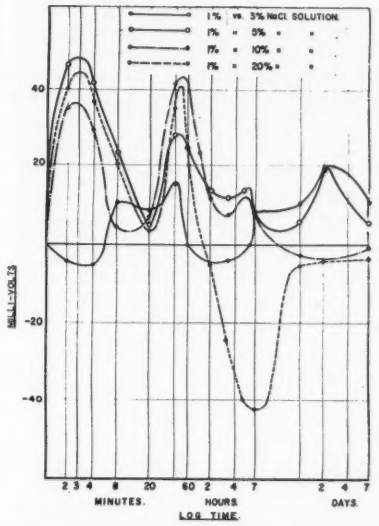

Figure 8—Differences in S. E. potential between steel in 1 percent NaCl solution and steel in solutions of higher salt concentrations.

3. Although variation in salt concentration of a neutral solution can affect the local cathode potential owing to changes in the oxygen solubility of the solution, this effect only becomes marked at relatively high salt concentrations.

Thus it is not expected that a cell of the type of No. 1 will provide any significant potential and that any current produced is therefore likely to be small.

Figures 7 and 8 illustrate the results obtained using the experimental half cells of this cell. In Figure 7, the single electrode potentials of the half cells are recorded and in Figure 8 the emf of the cell formed when each half cell is coupled to a one percent sodium chloride half cell. From these two figures it is clear that the steady or equilibrium potential reached some 24 hours after immersion does not change significantly with salt concentration and that potential differences are much smaller than those actually observed in corroding reinforced concrete.

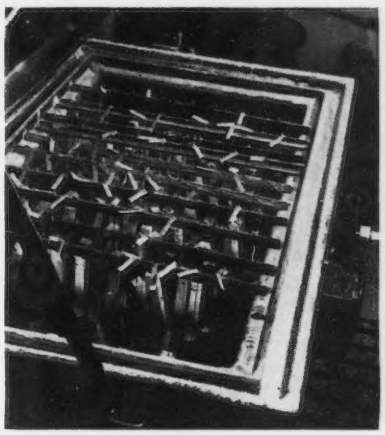

Figure 9—Specimens placed in position inside salt spray cabinet.

Cell No. 2 shown below corresponds to the case of steel embedded in concrete having both permeable and impermeable regions where carbon dioxide penetration at one point could lower the pH by carbonation. Although, theoretically, a potential difference of 0.25 to 0.45 volts could be anticipated from this cell, the conductivity of both electrolytes is small. In fact very feeble currents were obtained. In addition, the carbonate ion present in the anolyte could, under these conditions, act as an anodic inhibitor, thus further restricting the current.

Cell No. 2

Steel	Carbonated cement (low pH)	Alkaline cement (high pH)	Steel

Cell No. 3 shown below is an extension of cell No. 2 now allowing for salt penetration.

Cell No. 3

Steel	Carbonated cement plus salt	Alkaline cement plus salt	Steel

The steady potential of steel in aqueous cement extract (uncarbonated) is about —0.260 volts (saturated calomel). From Figure 7, the steady potential of the other half cell is —0.700 volts, and therefore the expected potential difference of a cell of this nature is —0.440 volts, which was found. Moreover, the presence of salt reduces the resistivity of the electrolyte, and currents of over 3 micro-amps per square centimeter may flow.

Cell No. 4

Steel (anode)	Permeable concrete low pH high Cl^-	Less permeable concrete high pH lower Cl^-	Steel (Cathode)

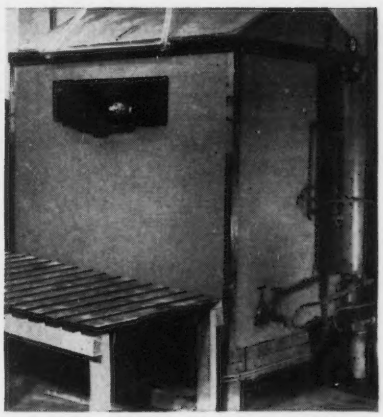

Figure 10—View of salt spray cabinet.

Cell No. 4 shown above is the general case of which the Cell No. 3 is the extreme condition and is considered to be the particular cell which is responsible for the corrosion of reinforcing steel in concrete described in this paper.

Cell No. 5

Steel	High oxygen availability	Low oxygen availability	Steel

The differential aeration cell shown above as Cell No. 5 will oppose the pH cell potential. But being of a smaller emf, it will be of less importance and Cell No. 4 will dominate.

Changes in Steel and Concrete Following Corrosion

During corrosion, chloride and hydroxyl ions diffuse towards the anode; cations such as those of calcium, sodium and ferrous will migrate toward the cathode. If, however, the solution is slightly alkaline or even neutral, ferrous ions will be deposited as ferrous hydroxide on the steel surface. During this precipitation, hydroxyl ions will be removed locally from the solution at a rate usually greater than that at which it is replenished by diffusion from the cathode. The solution will tend to form slightly acid conditions locally. Ferrous hydroxide will then take up oxygen and be converted into typical rust. As the hydroxyl ion concentration is reduced, precipitation of the ferrous hydroxide will occur progressively further away from the anodic area.

During periods when the moisture content of the concrete is high, electrical resistivity of the concrete is low, and the rate of anodic dissolution is high. In long periods of dry weather and consequent lower moisture content, the corrosion rate falls as the electrical resistivity increases while rust already produced tends to become dehydrated. Alternate wetting and drying during macro-cell corrosion produces the characteristic onion-skin type of corrosion product generally found in practice. As the amount of corrosion product increases, the internal pressure tending to crack the concrete increases until, with rupture and direct exposure to the atmosphere, conditions will change

and allow the usual micro-cell atmospheric corrosion to proceed at an accelerated rate.

Experimental Investigation of Corrosion Prevention Methods

In investigating the effects of various factors on the corrosion of steel in concrete, the electrical resistance method of Pletta et al[3] was considered but not adopted. Consideration also was given to the procedure of following single electrode potentials, but this was also discarded in favor of the more flexible weight-loss method used extensively in corrosion studies and also by Friedland[4] in a similar investigation.

Salt Spray Cabinet Tests

The principle of these tests was that a pickled, washed, dried and weighed steel rod could be placed in a mortar and the specimen exposed to an accelerated cycle of salt sprays and drying atmospheres at a temperature of 40 C in a salt spray cabinet. After an exposure of a definite period, the specimen could be removed and the steel rod extracted and pickled to remove corrosion products. A weight loss determination was made by re-weighing. The control specimens consisted of a mortar of fixed mix proportions, aggregate type, aggregate grading, curing procedure, water-cement ratio, compaction and cover of mortar over the steel. Each of these factors could be varied, keeping the remainder constant. Various treatments or additions also could be made to the control specification for more variations.

Figures 9 and 10 show the salt spray cabinet with the specimens in position; Table 2 gives some of the results obtained.

Discussion of Results

The control series of specimens consisted of 1 : 4 mortar with a water-cement ratio of 0.6. The aggregate was quartzite of a fixed grading and there was ⅜-inch cover of mortar around the steel. The procedure for compaction was kept constant throughout the series. The steel rods used were ¼-inch diameter and 4-inches long.

When the water-cement ratio was varied, the results showed a definite trend as illustrated in Table 2, Series b. High water-cement ratios resulted in a mortar which provided a less efficient protection to the steel than a mortar having lower water-cement ratio.

The same pattern of behavior can be seen in the results for variation of mix proportions (keeping water-cement ratio and aggregate grading constant). A richer mix, which is probably less permeable and contains a greater amount of free calcium hydroxide, provides better protection than a lean mix.

The results for surface coatings on the mortar, Series d in Table 2, demonstrate the extent to which protection can be achieved by excluding air, water and salt from the mortar. The salt spray cabinet tests give no indication of coating durability under actual exposure.

In Series e (aggregate), crushed aggregate was screened and recombined to provide the required control grading. Kruidfontein Sloot, a shrinking aggregate which is dimensionally unstable[11] when subjected to variations in moisture content, yields a mortar which, due to cracking, permits early corrosion to take place. Permeability of such aggregate probably adds to the severity of the corrosion. The behavior of the sound, inert aggregates, Norite and Quartzite is in contrast to this while Dwyka Tillite, which to some extent may behave similarly to Kruidfontein Sloot has also yielded a poor result.

From fundamental considerations of the corrosion of steel in concrete, it would be thought that the addition of a substance which could provide more alkaline material and lower the permeability by virtue of its small particle size would be beneficial. The addition of lime to the mortar (Series f in Table 2) confirms this supposition.

The data presented in Section g of Table 2 shows the beneficial effects of adequate curing; the effects of thickness of cover, Section h, illustrate the generally accepted advantage of increased cover.

Salt Spray Bath Tests

With the establishment of certain general trends, a further exposure program has been instituted in which reinforced concrete specimens will be exposed in an enlarged salt spray device shown in Figures 11 and 12. Duplicate specimens will also be exposed to natural conditions at an exposure site at Cape Town harbor.

Simultaneously, experiments will be conducted to determine the relative rate of salt penetration into concrete achieved by salt spray bath, salt spray cabinet and natural exposures at Cape Town and Durban.

Single Electrode Potential Observations

Single electrode or solution potential observations are of use in laboratory studies particularly in the action of inhibitors.

Work is in progress to determine the extent to which this method can be applied to establish whether steel in concrete is corroding, by measuring solution potentials on the concrete surface without damaging the concrete.

In this regard it is of interest to note the work of Stratfull,[6] recently reported, whose observations infer support of the corrosion cell proposed by Bird.[8]

The significance of pH and salt penetration in relation to cover may be seen in Figure 13. Samples of mortar drilled from a block previously used in a sea water penetration experiment, were analyzed for chloride and calcium hydroxide content. Single electrode potentials of the steel at the various depths of cover were

TABLE 2—Results of Salt Spray Cabinet Tests

	Specimen		Percentage Weight Loss After	
			9 Months	12 Months
a	Control Series (*)		1.2	1.8
b	Water cement ratio	0.95	2.2	—
		0.70	1.4	—
		0.60*	1.2	1.8
		0.45	1.0	0.9
c	Mix proportions (cement: sand by weight)	1:3	1.3	1.4
		1:4*	1.2	1.8
		1:5.5	3.7	Failure
d	Coatings	Silicone	Negligible	Negligible
		Tallow limewash	Negligible	0.1
		Bituminous	Negligible	0.1
		Others	0.6	0.7
e	Aggregates	Norite	—	1.2
		Quartzite*	1.2	1.8
		Dwyka Tillite	—	Failure
		Kruidfontein Sloot	Failure	—
f	Addition of 15% of lime, by wt. of cement	Magnesian	0.9	1.3
		Dolomitic	1.5	1.2
g	Curing (in humidity room)	21 days	0.7	1.0
		7 days*	1.2	1.8
		1 day	Failure	—
			Weight Loss in Milligrams Per Square Centimeter After	
			9 Months	12 Months
h	Cover of mortar (in inches)	⅜*	15	23
		5/16	35	48
		¼	29	69

* All series marked with an asterisk are control specimens.

Figure 11—Internal view of salt spray bath showing specimens in racks with spray nozzle in center.

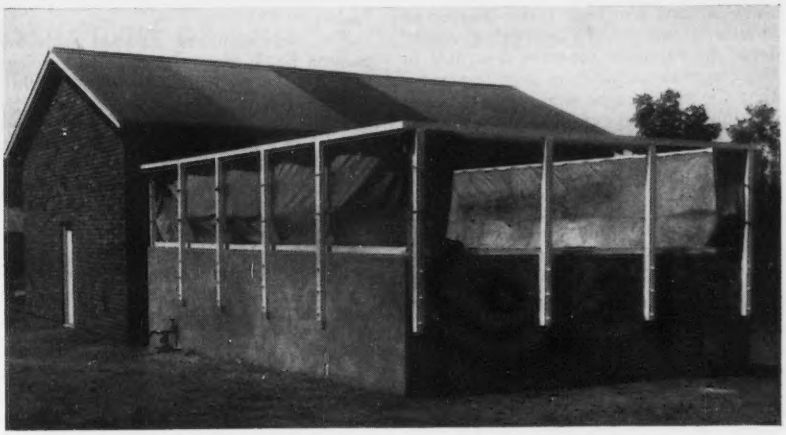

Figure 12—External view of salt spray bath with end canvas removed.

recorded from the previous experiment. When the block was broken, only the three top bars showed signs of corrosion; bars deeper than these were free from corrosion.

Methods of Corrosion Prevention

Corrosion of reinforcing steel, by whatever mechanism, is due primarily to the penetration of moisture, chlorides (sea salts) and air to the steel surface. Preventive methods must either prevent the penetration of these materials or render the reinforcement passive to their action.

Eight suggested methods of corrosion control are given below:

1. Location of Structure

The first precaution which could be taken to prevent corrosion of reinforcing steel, is to avoid the more aggressive locations where possible. There are obviously more important factors to consider when deciding the exact location of a bridge, for example, but if advantage could be gained by utilizing the natural shelter provided by some topographical feature in relation to the prevailing salt-bearing winds, this factor should merit serious consideration by surveyors and planners.

2. Design

Engineers could make an important contribution to corrosion prevention if their design excluded potentially dangerous features, such as scuppers which allow water collected on the structure to flow over concrete surfaces instead of being thrown clear. Rainwater from pools collected on the structure will leach out hydroxides from the concrete and accelerate carbonation; salty water flowing over the concrete surface could make salt available where none might occur normally.

Most authorities agree that provision of adequate concrete cover over steel is essential. A 3-inch minimum has been mentioned,[12] but 2-inch minimum is generally considered essential. Lastly, unnecessary reinforcing should be avoided, e.g. in hand rails or balustrading.

3. Concrete

Perhaps the biggest single contribution to the solution of this problem could be made by the concrete technologist. It would be trite to say that an impermeable concrete is the complete solution; however, by the application of the modern principles of concrete technology, it is possible to produce a relatively impermeable concrete which would certainly extend the life of most structures by a large factor.

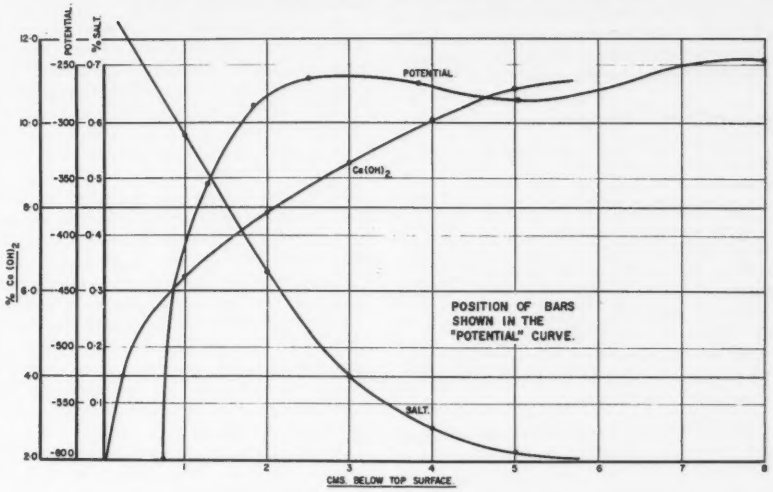

Figure 13—Curves showing pH, salt and incidence of rust.

Mair and Zietsman,[13] Shermer[5] and others have shown how important the control of water-cement ratio can be; other studies[3,4] have demonstrated the effect of factors such as aggregate grading, curing, richness of mix and compaction in making high quality concretes that preserve steel reinforcing. No matter how well prepared a specification may be, it requires careful supervision and site-control for it to be complied with in practice. This is not only the case in South Africa: Spencer[14] has commented in like fashion on structures in the United Kingdom.

4. Materials

Since the structures concerned are generally close to the sea, great care should be taken that the materials used in construction are free from salt. Water, both for mixing and for aggregate washing, and sand or aggregate used should be tested for salt. If salt is present, the aggregates should be well washed. Salt contaminated water should be rejected as unsuitable for construction work. Any additives for use in the mix should be carefully examined and should be avoided if they contain chloride.

The use of aggregates showing an abnormal dimensional change characteristic,[11] (shrinking aggregates) must be avoided. Aggregates that are permeable probably will yield concretes of higher permeability than would be the case with impermeable aggregates; hence the latter type should be used.

Ideally, the steel used should be clean and free from rust or millscale when the concrete is placed. Since this is generally regarded as an impractical recommendation and since rusty steel is reputed to result in a greater bond strength than clean steel, the only precaution which is likely to be taken in the matter of the steel is that before placing the concrete the steel be protected from marine atmosphere corrosion. If this is not possible, the steel should be well washed with fresh water when in position in the shutters to remove any salt deposits from its surface.

5. Surface Coatings

The use of certain coatings to prevent penetration of the corrosive materials into concrete is being investigated. That their application is beneficial is beyond question since their action is obvious;

their use has also been recommended in similar circumstance[6, 15] and initial results from the exposure program described in this paper are significant. It is important that the coatings be applied as soon as possible after the curing of the concrete; once salt is present in the concrete the effectiveness of the coating may be reduced.

So far, only methods aimed at preventing penetration of the aggressive materials have been mentioned. The following measures may also be considered:

6. Metal Treatment

In any treatment of the metal a prime consideration must be its effect on the strength of the bond between reinforcing steel and concrete. Reported experiments by overseas workers involving the use, for example, of galvanized steel for reinforcing, have yielded conflicting results, and it is planned to undertake bond strength tests for those methods of steel treatment considered efficacious in reducing or preventing the corrosion.

7. Cathodic Protection

The use of this technique to combat reinforcing steel corrosion has been considered.[6, 16] To ensure continuous economic protection, the cathodic metallic circuit should be electrically continuous if interference effects and anodic spots are to be avoided. Secondly, the electrolytic circuit should have a reasonably uniform resistivity which should not be so high as to necessitate excessively large voltages for providing the minimum current requirements. The following five considerations, therefore, merit attention:

(1) Electrical resistivity of concrete when dry may exceed 100,000 Ohm/cms[3] and when wet with salt water may be as low as 100 Ohm/cms.[3] Both these extremes could be met in the same structure under different weather conditions.

(2) Sacrificial anodes with their limited forcing potentials are unlikely to be satisfactory. Due to consideration (1) above, impressed current systems would require extensive anode arrangements if reasonable current spread were to be obtained, under dry conditions. Under wet conditions with the same voltage setting on an impressed current system, there would exist the serious risk of damage due to hydrogen evolution.

(3) Acids produced at the anode could cause chemical attack on the concrete.

(4) Alkali (sodium hydroxide) produced at the steel surfaces may also affect the concrete.[17]

(5) The operations involved in ensuring that all the reinforcing steel in a structure is electrically continuous are generally too extensive to be practicable.

From these considerations it can be seen that cathodic protection generally could not be applied economically to a new structure and is not a practical solution to the problem where it arises in existing structures.

8. Inhibitors

The steel surface coated with the gamma Fe_2O_3 film can act as an efficient cathode. If a non-conductive film of other oxides or hydroxides as produced by cathodic inhibitors, e.g. calcium carbonate, aluminium oxide, magnesium hydroxide, etc. could be deposited, the cathode efficiency would be markedly reduced. Unfortunately, adding soluble aluminum, zinc or magnesium compounds would result in their hydroxides being precipitated by the alkaline cement substances throughout the concrete mass and not selectively where they are required, viz. at the cathodic steel surface.

Anodic inhibitors such as alkalies, phosphates and chromates contain anions which either form sparingly soluble iron salts or form the gamma Fe_2O_3 film on the anodic steel surface, thus preventing ferrous ions from passing through and into solution at the anode.

This type of inhibitor is effective only when present in sufficiently high concentrations and is otherwise dangerous, because if added in insufficient quantities to stop attack, the corrosion becomes intensely localized and the attack is stimulated. Being anodic inhibitors, these substances are required at the anodic regions of the macro-cells on the steel surface and, when present in sufficient quantity, will inhibit corrosion by both macro- and micro-cell.

Unfortunately the following factors operate against the use of anodic inhibitors:

Should they be added to the concrete, they will be distributed evenly through the bulk of the concrete; if sufficiently high concentrations to be effective are used, the properties of the concrete may be affected adversely.

Should they be incorporated as a coating on the steel, the concrete-steel bond may be weakened.

The amount of inhibitor required to be effective increases as the chloride ion concentration increases. This may lead to the effective concentration of inhibitor for high chloride concentrations being inordinately high.

The solubility of the inhibitor should be such that while sufficient inhibitor is in solution to be effective it is not so great as to allow leaching out to take place.

From these considerations it is clear that until more fundamental work is done, the use of conventional inhibitors cannot be recommended at this stage.

Summary and Conclusions

Corrosion of reinforcing steel in concrete structures exposed to marine atmospheres is a major problem in South Africa and has also been reported to occur overseas.

Concrete, in which steel is normally passive, when penetrated by salt, water and air, allows certain micro- and macro-cell corrosion to proceed.

Investigation has demonstrated that externally applied coatings can reduce the corrosion significantly.

Theoretical and experimental evidence shows that the achievement of impermeable concrete, by the application of modern principles of concrete technology, and adequate cover are measures which can reduce corrosion.

Application of cathodic protection and the use of inhibitors are not recommended at this stage.

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Special mention must be made of the electrochemical studies of C. E. Bird (past Marine Corrosion Fellow attached to the C.S.I.R. Corrosion Unit of the National Chemical Research Laboratories) and the work of A. Gregor (past Marine Corrosion Fellow with the National Building Research Institute).

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Cathodic Protection of Lead Cable Sheath*

By W. H. BRUCKNER* and OLE G. JANSSON**

Abstract

The paper describes the results of laboratory tests with gradient lead cathodes in environments of high pH level. By observation of the corrosion limit and corresponding potential it was possible to define the cathodic protection requirements for permanence of metallic lead as a function of the pH of the environment. The increment of cathodic potential required for interface protection of partially immersed lead cathodes was also defined for laboratory test cells. The effects of overprotection and of a soil barrier to diffusion are shown in the paper to increase the susceptibility of the lead system to corrosion loss during periods of minimum current supply.

The instability of a lead electrode was evident in attempts to measure static potentials except under precisely controlled conditions not obtainable in the field. It was recommended, therefore, that potentials in the field be obtained with other standards than the lead electrode. 5.2.4

Introduction

IN PREVIOUS papers[1,2] it was indicated that lead could be given complete protection by cathodic means against corrosion in electrolytes of high alkalinity provided constant conditions of protective potentials were maintained. The present paper describes the constant conditions which must be attained to prevent corrosion as determined from gradient test cells. The data obtained in the research effort have shown that the corrosion of lead sheath experienced in the field is not due to cathodic corrosion but is due rather to failure of the cathodic protection system to supply adequate protection. It was not to be implied that cathodic corrosion of lead was not possible since the Cathodic Protection Laboratory has indeed confirmed its existence at current densities beyond about 30,000 ma per sq ft.

Pourbaix and Van Muylder[4] have presented a curve relating the threshold potential for the appearance of cathodic corrosion versus the pH. The relationship of pH and current density also shows that as the pH increases the current density required to initiate cathodic corrosion also increases and may go to values as high as 120,000 ma per sq ft. On this basis and on the basis of other experimental data cathodic corrosion was rejected as a mechanism for the service failure of lead cable sheath under cathodic protection. However, the consideration of cathodic corrosion has been prevalent for about 30 years and will be difficult to replace by the more appropriate terminology of "Corrosion by Cathodic Products."

Methods and Procedure

In the previous NACE paper by Bruckner and Lichtenberger[2] the concept of the gradient test cell and the gradient lead cathode was introduced. The term gradient was used to describe a variation in the protective potential over the length of the cell. The term gradient therefore was not used in its usual electrical engineering sense.

The objective of the gradient test cell was to supply a constant environment and to observe the incidence of corrosion and complete protection for the lead cathode as a function of the potential established along the lead cathode. It had been established previously that the pH of the gradient cell depended upon the current supplied and that diffusion in the absence of Ca or Mg ions would provide a uniform pH throughout the length of the 52 inch long cell after about two days of operation. For a current input of 3 ma the pH rose to about 9.5 and with 40 ma it rose to about 12.5. Thus it was not possible to select the pH of the environment independently of the current or the potential gradient. However, by initial adjustment of the pH and its maintenance during cell operation it was possible to operate four cells with an input of 3 ma and maintained at pH values of 11, 12, 13 and 14. It was expected that part of the lead cathodes most remote from the point of current entry to the cell would be subject to corrosion and would thus permit the establishment of minimum protective potential. For the cells with pH of 11, 12 and 13 the electrolyte was continuously circulated to provide a uniform pH throughout the length of the cell. For pH 14 it was considered unnecessary to resort to circulation. The form of the gradient cell was as shown in Figure 1 taken from reference 2. The electrolyte consisted of 50 ppm of NaCl in demineralized water to which NaOH was added periodically to maintain the desired pH. The cathodes were strips of lead rolled from cable sheath alloy to a thickness of 10 to 12 thousandths of an inch. The lead cathodes were partially immersed in order to study the corrosion behavior at the interface as well as below in the submerged area.

The above cells which are designated group A cells were operated for a total period of about 30 weeks during which time periodic observations were made of the extent of corrosion, pinholing, film formation and other aspects. The data obtained are presented later in the paper. A number of other gradient cells were set up and operated for periods of time shorter than 30 weeks in order to obtain additional data. Two cells were operated at 3 ma input with a gradient lead cathode fully immersed in the electrolyte, one at a pH of 13 and another at 14. Another gradient cell was operated at 1 ma input with a saturated Na_2CO_3 solution maintained at pH 13 with addition of NaOH. The cathode was partially immersed.

Another series of two gradient cells, designated group B, was operated with soil in contact with the gradient cathode. A relatively high current input of 40 ma was used for both cells and the soil was initially saturated with a solution of 6000 ppm NaCl and 4000 ppm of $CaCl_2$ in the

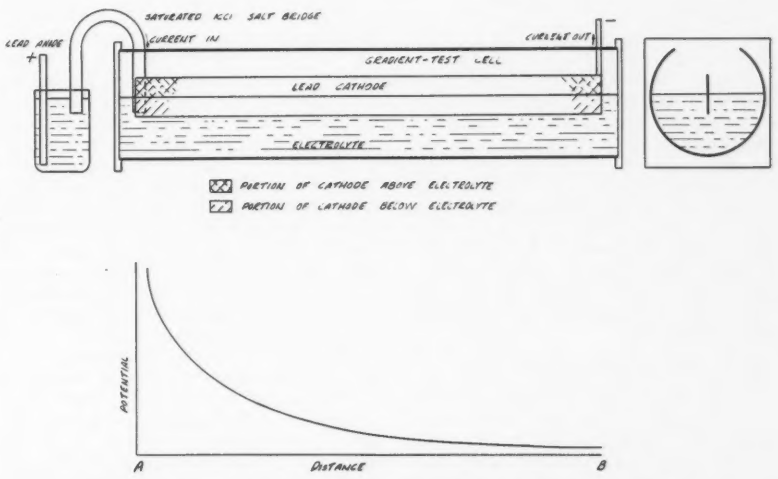

Figure 1—Schematic illustration of gradient test cell with 41-inch long lead cathode.

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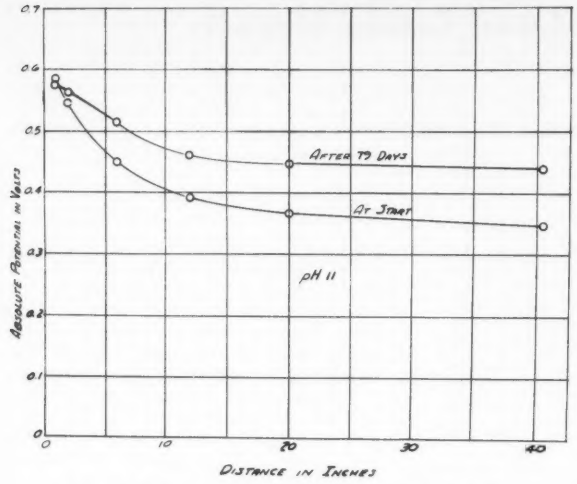

Figure 2—Potential versus distance for lead cathode at pH 11.

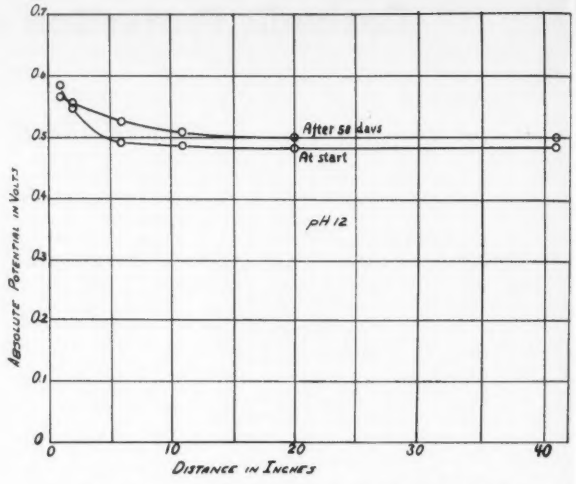

Figure 3—Potential versus distance for lead cathode at pH 12.

demineralized water. The cathodes were partially immersed in the soil saturated with the mixed salt solution. During operation of these cells demineralized water was added to maintain saturation. It was intended to operate one of the cells continuously and one intermittently with periods of one week on open circuit during which the lead strip would be without cathodic protection. The salt bridges through which the current entered the cell apparently were unstable at the 40 ma level. Thus the group B cells were in effect intermittently supplied with current when the salt bridge failed at any time during which the Cathodic Protection Laboratory was unattended.

Another phase of the research was an investigation of the conditions under which a stable static potential could be obtained for a lead electrode and to determine the potentials of lead under these conditions as a function of the environment pH. The data were to serve as a criterion for the continued use of a lead slug electrode for determining lead sheath potentials or its discard. The details of this phase of the research program are given in the sections on data and discussion.

Still another phase of the research was a survey of the pH of a cathodic protection system in relationship to the current input, the salt content and the volume of the electrolyte in the system. This series of tests was carried out with lead foil electrodes in separate anode and cathode beakers electrically joined by means of salt bridges. Periodic pH measurements were made to determine the change of pH with time for various constant values of current input, salt concentration and electrolyte volume. The experimental details and data are discussed below.

Data and Discussion

The gradient cells of group A operated at 3 ma current input and at pH of 11, 12, 13 and 14, schematically illustrated in Figure 1, were subjected to periodic measurement of the cathode potentials along the gradient and the condition of the surface of the cathode. The general experience with all these cells was the appearance of corrosion products on the surface of the lead cathode where the protective potential was a minimum (i.e., at point B of Figure 1). With increasing duration of operation of the cells the corrosion surface moved up on the gradient cathode toward point A. The thickness of the corrosion film decreased as the pH of the cell solution increased and became entirely protective first for pH 11 then later for pH 12. For pH 13 and 14 the film did not give protection against further corrosion. For pH of 12, 13 and 14 lead was deposited on the cathode in the region of point A. For pH 12 the deposition of lead ceased early in the operation history of the cell, due possibly to the complete protection provided in the less negative end of the cathode by the thick corrosion film. There was a slight decrease in the rate of lead deposition at pH 13 but at pH 14 there was no decrease. However, for the latter the deposit of lead was initiated quite late in the cell history.

The most important data was that for the pH 13 cell since the lead cathode developed pinholes in region B in 19 to 21 days of cell operation. Such a rapid rate of corrosion for the cathode strip would be equivalent to 1½ to 2 years of service life for a lead sheath having a nominal wall of 0.150 inch. For the cell at pH 14 the lead cathode corroded at a rapid rate but the attack was so uniform that the first pinhole occurred after only 51 days of operation. For both pH 13 and 14 cells the cathodes developed bands of red corrosion products just above the water line throughout the entire length of the 41 inch long cathode strips. The red band was wider and more prominent for the pH 14 cell.

The changes in the potentials of the gradient cathodes are shown in Figures 2, 3, 4, and 5 for various elapsed operating intervals. Figures 2 and 3 respectively for pH 11 and 12 and intervals of 79 and 58 days show a general increase in the potential due to several possible causes. The formation of a corrosion film is considered to have affected the potential by means of the added resistive component. The thicker film at pH 11 is thus reflected in the larger potential change in Figure 2 as compared with pH 12 in Figure 3. For pH 13 the data in Figure 4 show that after 102 days of cell operation the potential gradient is much more steep compared with its initial form. The deposition of lead at the high current end of the cell apparently had increased the surface area to such an extent that less current reached the more remote portion of the cathode and for this reason the potential became less negative. For pH 14, Figure 5 shows little change in potential after 65 days of operation, possibly due to the lack of film formation. The change which did occur however was a slight decrease in the negative potential the same as for the pH 13 cell. The large increase in surface area due to lead deposited had not occurred in the pH 14 cell at the end of 65 days of operation.

For all of the cells the aggressiveness of the alkaline solution in causing corrosion of the lead became less pronounced the longer the cells were operated. This was true to a maximum extent for pH 11 and 12, to a lesser extent for pH 13 and only slightly for pH 14. The reasons for the decreasing corrosion were considered to be the gradual absorption of CO_2 by the solution and the increasing concentration of plumbite ions in the solution. The paper by Delahay, Pourbaix and Van Rysselberghe[4] shows a minimum solubility of the $PbCO_3$ film at a pH of 9.5, thus at higher pH values the $PbCO_3$ film becomes more soluble and less protective. The latter paper shows also that between pH 5 to 11.5 if there is a sufficiently high CO_2 concentration the $PbCO_3$ film can provide passivity or immunity to corrosion without resort to cathodic protection.

From the observation of potential and corrosion behavior it was determined that at a pH of 14 the minimum protective potential was —0.59 volt to hydrogen to provide a life of about 18 to 20 years for a lead sheath with a 0.150 inch thick wall. For pH 13 this value was —0.54

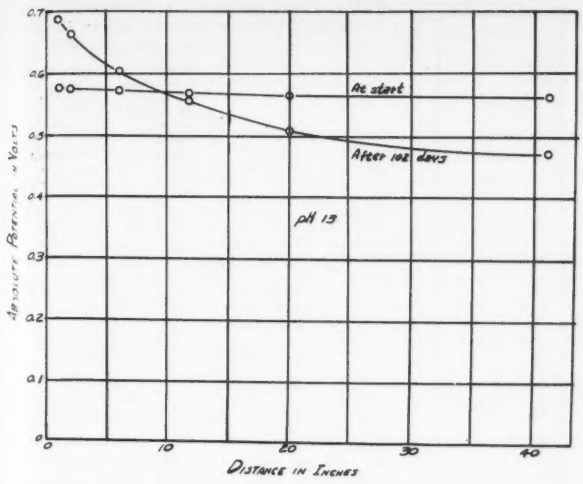

Figure 4—Potential versus distance for lead cathode at pH 13.

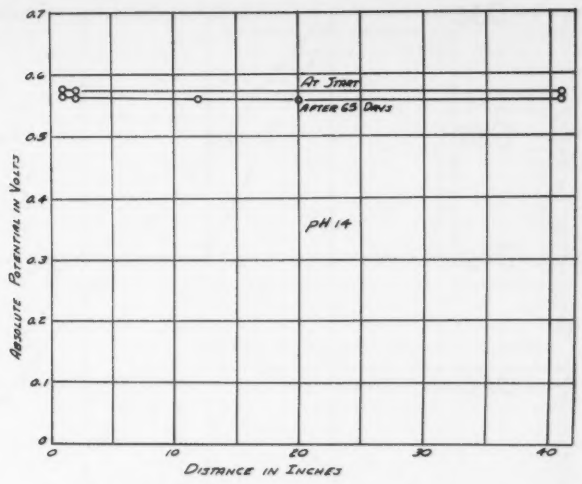

Figure 5—Potential versus distance for lead cathode at pH 14.

volt but for pH 11 and 12 this minimum protection could not be determined since the 3 ma input to these cells provided sufficient voltage at point B to prevent pinholing. Attempts were later made to obtain these potential minima for pH 11 and 12 by means of cells with lower current input.

For the two cells operated at pH 13 and 14 with the cathode totally submerged and with an input of 3 ma it was noted that the potential gradient was less steep. The corrosion rate at point B also was substantially less and there were no red or yellow colored corrosion films, all as compared with the partially immersed cathodes. For the cell operated at pH 13 and saturated in Na_2CO_3 the 1 ma input provided complete protection against corrosion or pinholing of the partially immersed cathode in contrast with the rapid pinholing previous experienced at pH 13 with solution initially low in CO_2 and with an input of 3 ma. The surface of the entire cathode appeared bright and free from corrosion products thus indicating the high level of protection offered by the $PbCO_3$ films when in contact with a saturated carbonate solution even at pH 13.

The data and observations on the two, group B, gradient cells will now be cited. These cells consisted of the gradient lead cathodes partially immersed in soil saturated with an aqueous solution of mixed Ca and Na chlorides. The current input to the cells was 40 ma and one of the cells was operated intermittently with intervals of one week of cathodic protection and then one week on open circuit. The other cell was operated on an intentionally continuous schedule. However, the salt bridge connecting the anolyte beaker to the gradient cell occasionally failed during periods when the Cathodic Protection Laboratory was unattended. Thus, instead of continuous operation, the cell had short overnight or week-end periods on open circuit.

Potential surveys along the gradient cathodes were not made for the group B cells but several surveys were made of the soil pH along the potential gradient. It was found that during operation at 40 ma the pH rose to a value of 13.5 to 14 at the high current end while at the low current end the pH ranged from 8 to 10. Evidently the soil is in an effective barrier to diffusion. Thus while the cell with solution only produced a maximum, uniform pH of 12.5 over the length of the cell this was not the case for operation in solution-saturated soil. It was expected that the lead cathode of the intentionally intermittent cell would corrode to some extent at the high current, high pH end during periods of open circuit. Thus this cathode was examined at frequent periods. It was noted that red corrosion products formed at the high current end in contact with the soil, although no particularly severe corrosion pattern was in evidence. Similar but infrequent inspection of the continuously operated cell showed some evidence of colored corrosion products at the soil air interface. After 12 weeks of operation the latter cathode was again removed and inspected. It was then found that a 1¼ inch long section at the high current end was so severely corroded as to leave the lead strip in a perforated, sieve-like condition where it had been most deeply imbedded in the soil. Evidently numerous, short periods of open circuit at pH 13 and 14 do more damage than longer periods in which the high pH may not be maintained.

In connection with the unexpected and rapid failure under soil condition it should be observed that for all of the gradient test cells the high current end of the cathode is characterized as being free from corrosion films. Over protection prevents the formation of protective corrosion films; thus upon cessation of cathode protection the film free area should exhibit a maximum initial corrosion rate.

The data and experimental observations in tests to determine the static potential of a lead electrode at different pH levels are described below. Initial attempts to obtain a stable potential for a lead electrode were unsuccessful for three conditions consisting of the following:

1. High purity lead wire immersed in alkaline solution.

2. High purity lead wire in solution of PbO in NaOH (carbonate free) and with bubbling hydrogen.

3. Lead plated Pt wire in previously deaerated solution of PbO in NaOH (carbonate free). Unsteady, non-reproducible potential values were obtained in all three cases and the potentials drifted continuously when measured against a saturated calomel electrode. A satisfactory procedure was found to be the use of a Pt electrode which was first plated with Cu then with Pb and used in a NaOH solution (CO_2 free) deaerated with streaming nitrogen bubbles. When the alkaline solution was saturated with PbO, stable reproducible potentials could be determined. The potentials obtained by means of this procedure are shown in the open circle points in the plot of potential versus pH in Figure 6. The experimental values may be compared with the solid line for the theoretical potential calculated from thermodynamic data with the use of the following equations:

(1) $E = 0.706 - 0.0886\ pH + 0.0295 \log a_{HPbO_2^-}$

(2) $\log a_{HPbO_2^-} = -15.68 + 1.00\ pH$ or combining (1) and (2) to give

(3) $E = 0.243 - 0.0591\ pH$

The dashed line in Figure 6 gives the locus of the theoretical potential calculated for a total lead concentration of 10^{-6} moles per liter (unsaturated solution) according to Delahay, Pourbaix and Van Rysselberghe.[3]

The two potentials of —0.54 and —0.59 volt representing the minimum protective potentials for respectively pH 13 and 14 which were obtained from the group A gradient tests are shown in Figure 6 as x's. These points fit the solid line curve closer than some of the experimental static potentials. Similar gradient test values for pH 11 and 12 were not available for comparison because of insufficient duration of the test schedule. However, on the assumption that the solid line curve adequately describes the

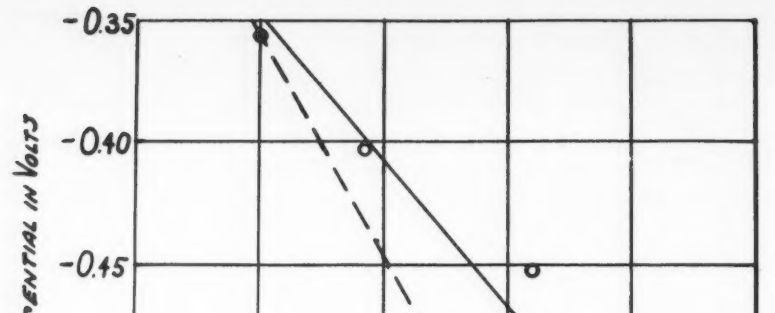

Figure 6—Potential versus pH for lead. Solid line is theoretical potential calculated from thermodynamical data. It corresponds to the potential of a lead electrode in saturated plumbite solution in the absence of carbonate and oxygen. Dashed line is for a total lead concentration of 10^{-6} moles per liter according to Reference 3. Open circles are from experimentally measured equilibrium values obtained in saturated plumbite solutions in the absence of carbonate and oxygen. The two points marked X at pH 13 and 14 are minimum protection potentials obtained from gradient test cells.

minimum protective potential for the full pH range as it does for pH 13 and 14, Table 1 has been prepared. The table refers to the service life of a cathodically protected lead cable sheath having a nominal wall thickness of 0.150 inch. The values given in the table assume that cathodic protection is uninterrupted and constant at the values shown.

The first column of values in Table 1 is for the case of a totally submerged cable sheath. The second column is for partial immersion conditions for which the gradient tests indicated that a potential about 10 millivolts more negative is required to prevent water-line corrosion than for the fully submerged sheath. The potentials required for cathodic protection in alkaline solution given in Table 1 will be modified by the effects of the concentration of CO_2. The maximum effectiveness of a $PbCO_3$ film in increasing corrosion resistance and in reducing cathodic protection requirement will exist for the low pH range.

Data and description are now given for the separate schedule of tests which were undertaken to clarify some aspects of the relationship between maximum pH and current input to a cathodic protection system. For an electrolyte containing only NaCl as the salt addition the maximum pH theoretically obtainable in electrolysis would be as follows:

Maximum pH	10	11	12	13	14
NaCl ppm	5.85	58.5	585	5,850	58,500

The above pH values are predicated on all of the NaCl being converted to NaOH and all of the Cl ions being converted to an insoluble precipitate or transported out of the catholyte. In an actual cathodic protection system the pH established will depend upon a number of other factors in addition to maximum salt content. The tests made in this part of the research program are given in the following.

Series 1 Tests

Series 1 tests which are summarized in Table 2 were made with two separate beakers each provided with a lead electrode and a saturated, KCl-agar salt bridge joining the two beakers. A 200 cc volume of electrolyte was placed in each beaker, the solution consisting of 100 ppm of NaCl in demineralized water. Separate runs were made with 0.1, 1, 4 and 8 ma input and the pH of the catholyte was determined at intervals of 2, 4, 24, 72 and 96 hours. These data are given in Table 2 where it will be noted that although the data are consistent with respect to increase in pH with time and current input the maximum pH is greater than is theoretically possible for a solution containing 100 ppm of NaCl. It must therefore be assumed that the unavoidable addition of KCl from the bridge was responsible for the excessive pH.

Series 2 Tests

Series 2 tests which are given in Table 3 were made in the same way as series 1 except that the volume of solution was one liter instead of the 200 cc previously used. Only two values of current input were investigated; 4 and 8 ma. The rate of increase in the pH of the catholyte is seen to be much less than for series 1 since with the same current the rate of removal of hydrogen ions or increase in the ratio of OH to hydrogen is the same but the product is distributed in a larger volume. The effect of the contribution of the KCl from the salt bridge is also less for the one liter volume.

TABLE 1—Potential—pH Relationship for Cathodic Protection of Lead Cable Sheath*

pH	Absolute Potential for Life of 20 Years or More for Full Immersion	Absolute Potential for Life of 20 Years or More for Partial Immersion
11	—0.41 volt	—0.42 volt
12	—0.48 volt	—0.49 volt
13	—0.53 volt	—0.54 volt
14	—0.59 volt	—0.60 volt

* 0.150 in. nominal wall.
Note: The above voltages are based on the hydrogen electrode voltage of zero. To obtain the voltage to the Cu/Cu SO_4 half cell add —0.32 volt to the values above.

Series 3 Tests

Series 3 tests are summarized in Table 4 and were made in the same way as series 2 except that the solution contained 1000 ppm of NaCl and only one current input of 8 ma was used. The results were the same as shown in Table 3 for 100 ppm NaCl and 8 ma thus indicating that the KCl bridge's contribution to the salt concentration predominated. The next series of tests were designed therefore to eliminate the effect of the bridge and to illustrate if possible the role of the transport of current by OH ions in the attainment of a stable maximum pH.

Series 4 Tests

Series 4 tests are summarized in Table 5 and were made with three separate beakers of one liter each of 100 ppm NaCl solution and a current of 8 ma. Lead electrodes were placed in two of the beakers to serve as separated anode and cathode. The third beaker was placed between the anode and cathode beakers and was electrically joined thereto by means of bridges containing a 100 ppm NaCl solution in agar. The data in Table 5 show that the pH of the central beaker first decreased then increased in 24 hours almost to the same value as that of the catholyte. The anolyte decreased to a stable value of pH 4.3.

The catholyte pH of 11.42 is just about the theoretical limit for 100 ppm of NaCl and the indications are that all of the Cl ions had been transported out of the cathode beaker in 24 hours. The initially large transport of current due to H ions is responsible for the decrease in the pH of the central beaker. The later and lower transport rate by Pb ions in combination with rising pH of the catholyte is responsible for the later increase in the OH ion concentration in the central beaker.

There are a number of inferences that can be derived from the pH current tests

TABLE 2—Relation of pH to Current Input for Anode and Cathode Volume of 200 cc Solution of 100 ppm NaCl

Test Duration, Hours	pH CURRENT IN ma 0.1	1	4	8
0	8.05	8.06	7.77	8.05
2	8.4	9.9	10.96	11.05
4	9.0	10.1	11.2	11.3
24	9.4	10.8	11.9	12.2
72	9.5	11.2	12.1	12.6
96	9.4	11.2	12.1	12.6

TABLE 3—Relation of pH to Current Input for Anode and Cathode Volume of 1000 cc Solution of 100 ppm NaCl

Time, Hours	CURRENT IN ma AT INDICATED pH 4	8
0	7.98	8.01
2	9.97	10.5
4	10.5	10.8
12	11.2	11.5
24	11.4	11.6
96	.....	11.5
120	11.4	11.5
168	11.5	11.6

TABLE 4—Relation of pH to Current Input of 8 ma for Anode and Cathode Volume of 1000 cc Solution of 1000 ppm NaCl

Time in Hours	pH
0	8.04
2	10.4
4	10.8
24	11.5
72	11.7
96	11.6
120	11.7
168	11.7

TABLE 5—Results of Test in Electrolysis of Three Compartment System with 1000 cc Solution of 100 ppm NaCl and Salt Bridges of 100 ppm NaCl Current 8 ma

Time in Hours	pH Cathode	Center	Anode
0	8.03	8.03	8.03
2	10.8	7.08	4.5
4	11.1	7.3	4.4
24	11.4	10.5	4.3

which have field application. The first is that the maximum pH attainable increases with NaCl concentration. However, the effects of concentrations of Ca and Mg ions and of CO_2 in the solution on the pH have not been examined. The second inference is in relation to the contribution of the salt bridge in increasing the pH above theoretical limits for the initial salt concentration at the cathode. Where the soil between anode and cathode has been saturated with de-icing salts it may have a contribution paralleling the authors' experimental experience in increasing the maximum pH of the water in the ducts.

The final observation on the above four series of tests is that it should be noted that the rate at which the pH increases is independent of concentration of NaCl but is dependent upon the current input in relation to the valume of solution surrounding the cathode. It would appear therefore that during a period of decreasing water level in the ducts the pH could increase to maximum level with low current input and could rise rapidly for large current input because of the reduced volume of duct solution.

Summary and Conclusions

It has been repeatedly demonstrated in the Cathodic Protection Laboratory that it is possible to provide lead with complete cathodic protection against corrosion in alkaline environment. In order to achieve such complete protection it is necessary to maintain constant conditions of protection with respect to solution level and a sufficiently negative potential in accordance with Table 1 and Figure 1 of the present paper. While the achievement of complete cathodic protection is demonstrable in the laboratory where the pH may be determined and controlled it will not be possible to attain such ideal protection in the field unless the pH is known at all points in the system and the potential everywhere on the lead cable sheath can be adjusted to equal or exceed (more negative) the values given in this paper.

It has been demonstrated in the laboratory that corrosion of lead can occur while under cathodic protection when the potential is not sufficiently negative to prevent corrosion in the environment. Corrosion of lead has also been demonstrated upon cessation of cathodic protection when the environment had a high pH resulting from previous cathodic protection.

With respect to the corrosion behavior of lead insufficiently protected by cathodic means in the range of 11 to 14 pH the following remarks apply:

(a) The corrosion loss by means of pinholing is a maximum for pH 13.

(b) The corrosion rate increases with pH from 11 to 14. Thus pH 14 has a maximum rate but due to more uniform corrosion than at pH 13, its rate of pinholing is less.

(c) The corrosion rate at pH 11 and 12 is low, relatively and may be reduced to still lower levels by formation of uniform protective films. The effectiveness of CO_2 in formation of protective carbonate films is high at these pH levels due to low solubility of the lead carbonate.

The relationships established for salt content, current input and electrolyte volume can be summarized as follows:

(a) The maximum pH which can be established in a cathodic protection system is dependent upon the salt content of the electrolyte surrounding the cathode and the salt available for diffusion into the system from the conducting medium between the anode and cathode.

(b) The maximum pH which is established is dependent upon the amount and distribution of the current received by the cathodic protection system and the opportunities for inter-diffusion along the system.

(c) The rate of increase of pH is governed by the current input, the ratio of current to electrolyte volume, the operating potential and the oxygen concentration. The lower the water level of a system the more rapidly the rise in pH with the same current input.

(d) The items a to c above are based on the presence of NaCl as the salt and the absence of calcium and magnesium as the salt cations. Where the latter are present they may modify the stated relationship of maximum pH, distribution of pH and the rate of rise of pH due to the precipitation of basic salts or of hydroxides.

The existence of a red film of PbO on lead during cathodic protection has been shown to be associated with conditions of partial immersion. The colored lead corrosion products form only at and above the interface of the partially immersed cathode. The colored corrosion products are considered to result from an environment of high alkalinity with insufficient cathodic protection current to prevent corrosion and insufficient solution access to remove the highly soluble PbO. The red corrosion products tend to form more readily at the interface of regions receiving maximum protection current. The existence of the colored products has been associated in the past with so called "cathodic corrosion." The Cathodic Protection Laboratory has demonstrated however that cathodic corrosion is possible only at such extremely high current densities as to be beyond any practical possibility. Instead the experimental evidence has supported the view that the corrosion experienced in the field is produced by the alkaline products of cathodic protection coupled with periodic reduction or loss of protection current.

The gradient tests have indicated that a substantial potential gradient in a cathodic production system on lead cable sheath promotes instability of the system. Over protection may have three major effects as follows:

(a) Effect a high pH locally, especially with soil or salt or with low solution level.

(b) Effect a deposition of lead to further increase the potential gradient and reduce protection in other parts of the system.

(c) Prevent formation of protective

films thus providing minimum corrosion resistance at maximum pH upon reduction of cessation of cathodic protection.

The data from the gradient tests thus indicate that failure of lead cable sheath can occur in underprotected as well as in overprotected regions.

In the laboratory where constant current to a gradient test cell could be maintained, an increase in the salt concentration had the advantage of providing more uniform distribution of protective current. This resulted in lesser lengths of both over protected and insufficiently protected cathode areas. In the field a cathodic protection system ordinarily receives current in accordance with the conductivity of the medium between anode and cathode. Thus the independence of current and conductivity available to laboratory control cannot be exercised. In view of the increasing amount of deicing salts applied to streets the economic feasibility of instituting current control independent of salt content should be investigated.

The gradient tests have indicated that a partially submerged lead cable sheath of nominal 0.150 inch wall thickness could be penetrated by pinholing in a period of two to five years under certain constant conditions of low level cathodic protection and pH beyond a value of about 12. The actual service life of cathodically protected lead sheath will vary with the integrated effects of the time during which the sheath is given less protection than required for a specific environment. The fully immersed cable sheath has many advantages in cathodic protection over one partially immersed or one which alternates between the two conditions. For the fully immersed system, the accelerated interface attack is absent, the current distribution is more uniform and available to lower potential regions in a gradient, the pH increases at a lower rate for the same current input to the system and the formation of more protective carbonate films is probable rather than the more soluble, less protective, colored oxide films at the interface.

Reports from the field confirm the experimentally anticipated general reduction in corrosion problems for continually and fully immersed lead sheath cathodically protected even where salt content of the environment is high. It is therefore recommended that an analysis be made of the costs of duct design and pumping requirements to provide full immersion for cable sheath as compared with costs of furnishing neoprene or other jacketing and the costs of system failure and replacement.

The difficulties which were associated with development of reproducible potential measurements for lead strongly suggest that the use of a lead slug for field measurements of cable potentials be discontinued. Since stable, reproducible potentials for lead at different pH levels could be obtained only in the absence of oxygen and CO_2 and only when the solution was saturated with $PHbO_2^-$ it will be obvious that field conditions cannot provide a stable potential for the lead slug. Moreover even under ideal conditions it would be necessary to determine the pH of the environment of the lead slug before any measured potential could have significance. The use of a standard half cell based on copper or silver is recommended instead of the lead slug.

The information developed by the cathodic protection research program should make it more easily possible to assign causes to current failures of lead cable sheath and to provide corrective measures. Field surveys could more readily anticipate regions of failure and local need for corrective if they could provide information on variation in potential, pH, salt content, silt access and solution level for each duct run. It is recommended that an effort be made to develop methods of determining locus of maximum pH and its value and the potential gradients. The possibility of using an antimony electrode coupled with a silver/silver chloride half cell for field pH measurement has been considered by the Cathodic Protection Laboratory. Exploratory work is required to develop the constants for such measurement and to provide a cell of sufficiently small size for use in occupied ducts.

Acknowledgments

The Commonwealth Edison Company of Chicago through its Edison Research Committee sponsored the research program and contributed helpful interest and guidance. Undergraduate assistants, Harry Maleski and Dallas Pasley performed some of the control tests necessary for the conduct of the research program.

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2. W. H. Bruckner and W. W. Lichtenberger. Cathodic Protection of Lead Cable Sheath in the Presence of Alkali from Deicing Salts. *Corrosion*, 14, 165t-170t (1958) April.
3. P. Delahay, M. Pourbaix and P. Van Ryselberghe. Potential pH Diagram of Lead and Its Applications to Study of Lead Corrosion. *J. Electrochem Soc.*, 98, 57 (1951).
4. J. Van Muylder and M. Pourbaix. Corrosion et Protection Cathodigues du Plomb. Rapport Technique No. 14 Cebelcor.

Any discussion of this article not published above will appear in the December, 1959 issue

New Technical Committee Officers Elected

Bigos

Bloom

Peabody

Volkening

Dorsey

Emerson

Wasson

Hochman

Hoy

Hugo

Keane

7 Task Groups Proposed At T-2M Chicago Meeting

Seven new task groups were proposed for approval at the Chicago meeting of Technical Unit Committee T-2M, Aluminum Pipeline Corrosion. Officers elected were Chairman H. P. Godard, Vice Chairman R. S. Dalrymple and Secretary C. F. Warnock.

Five Task Groups Make Reports At T-6D Meeting

Reports from five task groups were given at the Chicago meeting of T-6D Industrial Maintenance Painting.

T-6D-1, Economics of Maintenance Painting, summarized the results of questionnaires. The report was an analysis of trends leading to either increasing or decreasing cost in maintenance painting and was prepared by a study of individual replies.

T-6D-2, Standardization of Scope of Painting Specifications, report on specifications has been forwarded to the group committee chairman for disposition.

T-6D, Paint Programs, gave a preliminary report and asked for criticism on the industrial maintenance painting program survey.

T-6D-4, Specifications for Shop Cleaning and Priming, gave a short review on preparation of specifications for shop priming and coating. A suggested outline is to be completed before the next meeting in Denver.

Exploratory Task Group T-6D-5 on Painter Safety and Painter Education has been investigating the need for future work on painters' safety and education. By commitee action, this group was divided into two task groups: one to pursue the safety program and the other painter education.

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7464 copies of NACE Technical Committee Reports published in CORROSION were sold in 1958.

1959-61 Chairmen Elected for 1 Group, 5 Unit Committees

New chairmen and officers were elected recently to T-2B, T-3F, T-7, T-2C, T-6G and T-9B Committees.

T-2B

Chairman of T-2B, Anodes for Impressed Currents, is R. J. Emerson, corrosion engineer with El Paso Natural Gas Co., El Paso, Texas. Two other officers were re-elected: Vice Chairman J. S. Dorsey, corrosion engineer with Southern California Gas Co., Los Angeles, and Secretary L. A. Hugo, manager of Phillips Petroleum's Corrosion Survey Branch at Bartlesville, Okla.

T-3F

Officers for T-3F, Corrosion by High Purity Water, were re-elected. They are Chairman R. U. Blaser, Research Center, Babcock & Wilcox Co., Alliance, Ohio, and Vice Chairman M. C. Bloom, Metallurgy Division, Naval Research Laboratory, Washington, D. C.

T-7

Paul C. Hoy was elected vice chairman of Technical Group Committee T-7, Corrosion Coordinating Committee. He is associated with the Dayton Power & Light Co., Dayton, Ohio.

T-2C

L. C. Wasson, a director of the research and development division of A. O. Smith Corp., Milwaukee, Wis., was elected chairman of T-2C, Criteria for Cathodic Protection. New vice chairman is A. W. Peabody, electrical engineer with Ebasco Services, Inc., New York City.

T-6G

New officers for T-6G, Surface Preparation for Organic Coatings, are Chairman Joseph Bigos, associated with the applied research laboratory at United States Steel Corp., Monroeville, Pa., and Vice Chairman John D. Keane, who is director of research and executive secretary of the Steel Structures Painting Council, Pittsburgh, Pa.

T-9B

Officers recently elected for T-9B, Preservatives and Their Appraisal, are Chairman Harry Hochman, director of the Chemistry Division, Research Department, U. S. Naval Civil Engineering Laboratory at Port Hueneme, Cal., and Vice Chairman V. B. Volkening, group leader in materials evaluation at Dow Chemical Co., Freeport, Texas.

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226,800 copies of 25 NACE Technical Committee Reports were distributed in 1957. In 1958, 210,300 copies of 12 NACE Technical Committee Reports were distributed.

15 Guest Speakers Lead Discussions At T-5C-1 Meeting

Fifteen speakers discussed various phases of cooling water corrosion at the June 9 meeting of T-5C-1, Corrosion by Gulf Coast Cooling Water (South Central Region), held at the Houston Engineers Club, Houston, Texas. Speakers were from the Gulf Coast area, Philadelphia, Chicago, Cleveland, New York City and Wilmington, Del. Total attendance was about 70.

This meeting was one of the best held by the committee, according to members present.

Speakers and Discussion Topics

Speakers and their topics for discussion were as follows: Introduction and Review by J. M. Brooke of Phillips Petroleum; Solid Coupon Evaluation by R. V. Comeaux of Humble Oil & Refining; Experimental Exchangers by W. W. Wheeler of Rohm & Haas; Corrosometer by J. M. Brooke; Introduction to Fouling Rates vs. Velocity of Water by W. B. Thomas of Texaco Inc., Contribution by Betz, Inc., by C. W. Brown; Introductory Remarks on Performance of Side-Stream Filters by J. M. Brooks; Use of Floc Agents by J. B. Cruel of Nalco Company; Substitutes for Chlorine by B. F. Shema of Betz, Inc.; Chlorine Residual Recorders by S. H. Newland; Back-End Chlorine Injection by R. V. Comeaux; Plastic Fill by J. W. Wilson of Fluor Products Co.; and Ceramic Towers by R. S. Mercer of Gulf States Engineering.

Roundtable Discussions Held

Two roundtable discussions were included: one by J. J. Picarazzi of Cities Service on the performance of steel tube bundles and one by W. G. Ashbaugh of Union Carbide on stainless steel performance and other tube materials.

An open discussion on bundle pretreatment was also held.

A short business session followed the meeting. A nominating committee was appointed to select officers for election. When elected, the new officers will meet with the outgoing officers to plan future T-5C-1 activities.

T-1F Reorganizes Two Task Groups

Technical Unit Committee T-1F, Metallurgy, reorganized two of its task groups at a meeting held during the March Chicago conference.

Numerical identities of the groups were retained but their functions were modified and broadened. T-1F-1 was designated as Tubular Goods Corrosion Metallurgy and T-1F-2 as Sucker Rods Corrosion Metallurgy.

Because the groups were to be working groups, membership was held to a maximum of seven. Membership for the two groups is given below:

T-1F-1: W. F. Oxford, Jr., chairman, R. A. Prange, Rado Loncaric, John Fraser, M. Holmberg, P. D. Thomas and R. C. Bowden.

T-1F-2: W. C. Koger, chairman, F. A. Prange, F. L. Current, J. Mason, H. Cooley and G. E. Flavell.

Topics discussed during the meeting included alternate string sucker rod testing, alternate string coupling tests and monel tubing and sucker rod tests.

Next T-1F meeting is planned tentatively during the 1959 South Central Region Conference in Denver.

T-4B Reorganization Approved at Chicago

Reorganization of Unit Committee T-4B, Corrosion of Cable Sheaths, was approved at the meeting of the Technical Practices Committee during the Chicago conference.

Five task groups of T-4B were designated as unit committees under T-4, Utilities. The new designations are as follows:

T-4B-1 becomes T-4B; T-4B-5 becomes T-4C; T-4B-4 becomes T-4G; T-4B-3 becomes T-4H and T-4B-2 becomes T-4J.

Work of Task Group T-4B-6, Stray Current Electrolysis, was proposed to be assigned to task groups under appropriate unit committees.

Re-organization of T-4B was proposed so that work of the task groups representing the common interests of the power and communications industries could be accomplished more expediently and be given suitable recognition.

Down Hole Pump Corrosion Discussed at T-1D Meeting

Down hole pump corrosion and future activities were discussed at the Chicago meeting of T-1D, Sour Oil Well Corrosion.

Although rod and tubing failures seem to be controlled by the use of inhibitors, pump failures have not been satisfactorily controlled. Two areas of interest on the problem were discussed at length: a cooperative pump testing program similar to the mixed string sucker rod testing conducted in the past, and a cross-reference or catalog of trade names and alloy designation.

T-2 Hears Unit Reports

Reports from 8 unit committees and 12 task groups were heard at the meeting of Group Committee T-2, Pipe Line Corrosion, held during the Chicago conference.

Unit committee reports were from T-2B, T-2D, T-2E, T-2F, T-2G, T-2H, T-2J and T-2K.

T-5A-1 Compares Materials

Task Group T-5A-1, Corrosion by Sulfuric Acid, held a general discussion on comparative use of steel, cast iron and ductile iron piping and valves in strong sulfuric acid at its meeting during the Chicago conference in March.

Consensus of opinion was that steel and cast iron were about equal in static service performance, but cast iron was preferred for flow conditions. Ductile iron was considered to have equal but not necessarily superior resistance to cast iron.

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Fifteen local corrosion control committees have affiliated with NACE's T-7 committee in two years.

New Cooling Water Task Group Forms

A new task group on cooling water corrosion problems to be called T-5C-2 Corrosion by Cooling Water (North Central Region) will hold its organizational meeting during the 1959 North Central Region Conference, October 20-22, in Cleveland, Ohio.

This task group will consider any phase of corrosion fouling or scaling caused by cooling water. Typical problems for the group's consideration will be methods of feeding and controlling acid injection, where to inject acid, where to sample stream, problems of cooling tower deterioration, standard methods of evaluating water treatment programs and standards of equipment design to avoid galvanic couples.

Persons engaged in this work are invited to become members of the new group and to participate in its organization at Cleveland. Time will be given to discuss individual water corrosion problems brought up by each member.

Additional information can be obtained from William A. Hess, Standard Oil Company of Ohio, Midland Bldg., Cleveland 15, Ohio.

Tests and Reports Given At T-5E Chicago Meeting

Stress corrosion cracking tests and reports were the main topics for discussion at the meeting of Unit Committee T-5E, Stress Corrosion Cracking of Austenitic Stainless Steel, held in Chicago during the annual conference.

M. E. Holmberg reported on the status of the joint ASTM-NACE task force on stress corrosion cracking. He stated that the report is about 75 percent complete.

A discussion was held on the types of corrosion coupons used in various plants and laboratories.

Ship Hull Corrosion Discussed by T-3G-1

Discussions involving members' experiences with ship hull corrosion and successful control measures used were featured at the Chicago meeting of Task Group T-3G-1, Cathodic Protection of Hull Bottoms of Ships.

Three topics were included in the discussions: cathodic protection of stern areas of hulls, cathodic protection of shafts and propellers and types of reference electrodes, their positioning, interpretation of readings, etc.

Steel Use Is T-4A Topic

Use of steel for grounding and compatability of steel and copper in the ground were discussed at the Chicago meeting of T-4A, Effects of Electrical Grounding on Corrosion. Fifty-one members and guests were present.

Members discussed their companies' experience in electrical grounding and answered individual questions presented.

NACE NEWS

Half of Booths Sold for 1960 Corrosion Show

68 Booths Are Sold; Plans, Information Are Still Available

Sixty-eight booths, over half the total available, have been contracted for the 1960 Corrosion Show to be held March 15-17 in conjunction with the 16th Annual NACE Conference in Dallas, Texas.

The Corrosion Show will be held in the Dallas Memorial Auditorium. Technical meetings for the annual conference will be held on the two floors above the exhibit area. Stairs at each end of the exhibit area lead to these rooms.

8 x 10 Booths

All booths will be 8 feet deep and 10 feet wide with necessary connections for water, drainage, electricity and air. Booths have been arranged to give best traffic flow to as many booths as possible.

A descriptive brochure giving floor plan of the booths and pertinent information was mailed to prospective exhibitors in May. Copies of the brochure are available from R. W. Huff, Jr., NACE Exhibits Manager, 1061 M & M Bldg., Houston 2, Texas.

Products and Equipment to be Exhibited

Products of interest to corrosion engineers and supervisory personnel involved in corrosion control to be shown at the Dallas Corrosion Show include the following:

Aluminum, stainless steel and wrought iron products, protective coatings and linings, plastic products, cathodic protection equipment, pipe and tubing products, blast cleaning equipment, filters, valves, pumps, corrosion survey equipment, gaskets, seals, insulation, paints and primers, corrosion inhibitors, instruments, process equipment, tape coatings, lubricants and painting equipment.

List of Exhibitors

Those companies which have contracted for booths are listed below.

Aluminum Company of America
Amercoat Corporation
American Smelting & Refining Co.
Armco Steel Corporation
Brance-Krachy Co., Inc.
A. M. Byers Company
Carboline Company
The Carpenter Steel Co.
Cathodic Protection Service
Central Plastics Co.
Clementina, Ltd.
Corrosion Rectifying Co.
COSASCO Div., Perfect Circle Corp.
John L. Doré Co.
Fibercast Co.
R. C. Foltz Company
The Garlock Packing Co.
The Glidden Co.
Gulf States Asphalt Co., Inc.
Johns-Manville Sls. Corp.
Lincoln Engineering Co.
F. H. Maloney Co.
Minnesota Mining & Mfg. Co.
National Carbon Co.
Orchard Paper Co.
Otis Engineering Corp.
The Pfaudler Co.
Plicoflex, Inc.
Radio Receptor Co., Inc.
Reilly Tar & Chemical Corp.
Resistoflex Corp.
Royston Laboratories, Inc.
Shell Chemical Corp.
Shell Oil Company
Socony Paint Products Co.
Standard Magnesium Corp.
Tejas Plastic Materials Supply Co.
Texsteam Corp.
Truscon Laboratories
The U. S. Stoneware Co.
Visco Products Co., Inc.

Southeast Region News

Oct. 1-2 Conference Speakers to Include President Godard

NACE President Hugh P. Godard of Aluminum Laboratories, Ltd., Kingston, Ontario, Canada, will be one of the speakers at the 1959 Southeast Region Conference to be held October 1-2 at the 1959 Southeast Region Conference to be held October 1-2 at the Robert Meyer Hotel in Jacksonville, Fla. He will speak on "What NACE Can Do For You."

Other speakers and their topics to be included are listed below.

Economics of Maintenance Painting, Kenneth Tator of Kenneth Tator Associates, Coraopolis, Pa.

Rigid Plastic Pipe, W. R. Dana of Amercoat Corp., South Gate, Cal.

Salt Water Corrosion in Refineries, Paul J. Hurd of American Oil Co., Yorktown, Va.

Aluminum in the Pulp and Paper Industry, Harry Fritts of Aluminum Company of America, New Kensington, Pa.

Biological Deterioration of Materials in Marine Environments, Albert P. Richards of William F. Clapp Laboratories, Inc., Duxbury, Mass.

Other speakers to be scheduled for the conference, according to George M. Jeffares, general program chairman, will speak on corrosion problems in the marine, pipe line, pulp and paper, refinery and textile industries.

A business luncheon, social hour and annual banquet have been scheduled for the first day of the conference.

A final program of the conference will be published in the August issue of CORROSION.

Tidewater Section Secretary-Treasurer Otis J. Streever, corrosion engineer at the Newport News Shipbuilding and Dry Dock Co., has retired after 40 years with the company. His career has been in the field of cathodic protection of ships. In addition to NACE, he holds membership in AIEE and the Virginia Tidewater Corrosion Control Committee.

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Birmingham Section heard R. A. Davis of Chicago Bridge and Iron Company present a paper titled Hortonclad at the June 5 meeting. Discussion was held on the possibilities of the section setting up a short course in the near future.

Canadian Region News

Edmonton Section had M. E. Parker, corrosion consultant, as guest speaker at the May 28 meeting. His topic was Snares and Pitfalls in Cathodic Protection.

SECTION CALENDAR

July
30 Teche Section. Petroleum Club.

August
27 Teche Section. Petroleum Club.

•

NACE's 16th Annual Conference and 1960 Corrosion Show will be held March 14-18 in the Memorial Auditorium, Dallas, Texas.

Technical Topics To Be Included In December Index

Beginning with the index to Volume 15 of CORROSION to be published in December and included in the December issue, articles in the Technical Topics Section will be included in the alphabetical subject index, accumulated contents, and their authors listed in the alphabetical author index.

Persons who customarily extract Technical Section pages from the volumes of the issue for binding are reminded that it will be advisable to extract also the pages contained in the Technical Topics Section. If these are added to the pages of the Technical Section from each issue it will be relatively easy to locate the articles.

Regional Conferences

Final programs of the Southeast, Northeast and Western Region Conferences and Advance programs of the South Central and North Central Region Conferences will appear in the August issue of CORROSION.

HOUSTON SECTION PICNIC held May 12 at the Texaco Country Club was attended by about 200. Section members' families and guests were invited. Activities included golf, swimming, dancing and a barbecue dinner.

Muehlhause, Paisley Join in Region's Education Activities

Muelhause

Paisley

Two persons have been added to the South Central Region Educational Committee. They are Gerald J. Paisley, who will be the Kansas Representative on the committee, and Edwin H. Muehlhause, chairman of the North Texas Section Educational Committee.

Mr. Paisley is corrosion engineer with Mobil Oil Company's Central Pipe Lines Division in Wichita, Kansas. An active NACE member for seven years, he has a BS in electrical engineering from Kansas State College.

Mr. Muehlhause, associated with Lone Star Gas Co., Dallas, Texas, has been an NACE member for eight years. He has a BS in chemistry.

In its efforts to encourage high school students' interest in science and corrosion engineering projects, the South Central Region Educational Committee has established contact with the Science Clubs of America and Future Scientists of America Clubs, according to John Loeffler, committee chairman.

The Rocky Mountain Section Educational Committee invited the winner of the Physical Science Group of the Colorado-Wyoming Regional Fair for Future Scientists of America Clubs to be a guest at the section's May meeting. The student's teacher was also invited. An award of $50 in books was made to the student winner and a similar recognition award to his school library.

Advance program announcements of the Oct. 12-5 South Central Region Conference in Denver will be published in August issue of CORROSION.

South Central Region News

East Texas Section heard John Goforth, sales manager for Pipe Coat Service Corp., speak on Pipe Line Process of Internal Cleaning and Coating of Buried Pipe Lines at the May 26 meeting.

•

Permian Basin Section will discontinue monthly meetings until September. The summer months will be devoted to committee meetings to prepare the 8th Biennial Section Corrosion Tour.

Barney Goodman of Tube-Kote, Inc., presented a paper on Polyurethanes—Plastics with an Oil Field Promise at the May 12 meeting.

•

West Kansas Section had two South Central Region officers as guests at the May 7 meeting: J. A. Caldwell, regional chairman, and Dan H. Carpenter, regional secretary-treasurer.

Mr. Caldwell also was program speaker on Elements of Corrosion Inhibition.

Officers of the section are Roy Junkins, chairman, and Ray E. Walsh, vice chairman.

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Corpus Christi Section saw a pictorial presentation of common types of corrosion associated with the refining, storing and handling of crude oil and finished petroleum products at the May 26 meeting. The presentation was made by Chuck Allison, chemical engineer with Southwestern Oil & Refining Company.

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Teche Section heard John Rogers, Amercoat Corp., Houston, Texas, speak on inorganic permanent primers at the May 21 meeting.

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Central Oklahoma Section had J. E. Landers of Continental Oil Company as guest speaker at the May 11 meeting. He spoke on well casing corrosion in Conoco Allphin Pool in Kansas.

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Thirteen technical committee meetings are scheduled for the South Central Region Conference, October 12-15, in Denver, Colorado.

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South Central Region's 1959 Conference will be held October 12-15 at the Cosmopolitan Hotel in Denver, Colo.

•

News Deadline for CORROSION is the 10th of each month.

Houston Section held a panel discussion on corrosion by Gulf Coast cooling waters at the June 9 meeting. Panel members included R. P. Gulley of Gulf Oil Corp., J. M. Brooke of Phillips Petroleum Co., A. S. Krisher of Monsanto Chemical Co., R. V. Comeaux of Humble Oil and Refining Co., W. G. Ashbaugh of Union Carbide Chemicals Co., and Moderator Marshall Elliott of Gulf Oil Corp.

New section officers elected are Chairman C. B. Tinsley of Southwestern Bell Telephone Co., Vice Chairman O. A. Melvin of Carboline Co., Secretary-Treasurer J. L. Weis of Diamond-Alkali Co., and Trustee J. T. Payton of Texaco Inc.

Shreveport Short Course Chairmen Are Appointed

Committee chairmen have been appointed for the 9th Annual Shreveport Section Short Course to be held September 22-23 at Centenary College. They are as follows:

General Chairman Ned C. Stearns, Vice Chairman and Program Chairman Jack R. Walker, Publicity Chairman James Butler, Finance Chairman Dickran Tefankjian, Entertainment Co-Chairmen Frank Therrell and Sam Jones, and Registration Chairman Gene Moore.

100 Attend Teche Section Corrosion Short Course

A total of 100 persons registered for the 3rd Annual Corrosion Control Short Course held June 2-4 at Lafayette, La. Industries represented by the registrants were 32 from oil producing companies, 15 from pipeline companies, 45 from oil service companies, 4 from colleges and 4 from utilities.

Fifty-four NACE members and 46 non-members attended, according to John Selleck, short course director affiliated with Galloway Pipe Guard.

The short course is sponsored annually by the Teche Section and Southwestern Louisiana Institute.

SOME OF THE SPEAKERS at the 3rd Annual Corrosion Control Short Course sponsored by the Teche Section and Southwestern Louisiana Institute are shown above. From left to right, they are W. M. Kelly of Otis Engineering Corp., Dallas, B. F. Davis, Jr., of Atlas Powder Co., Houston, J. R. Oliver, H. H. Fischer of Nocor Chemical Co., Odessa, R. M. Robinson of Continental Oil Co., Houston, D. R. Fincher of Tidewater Oil Co., Houston, R. C. Booth of Plastic Applicators, Inc., Odessa, W. R. Nelson of Texas Pipe Line Co., Houston, J. G. Davis of Cathodic Protection Service, Houston, and Arthur Uhl of Southwestern Louisiana Institute. The short course was held June 2-4 on the institute campus.

NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1959

Sept. 29-30, Oct. 1—Western Region Conference. Bakersfield Inn, Bakersfield, Cal.

Oct. 1-2—Southeast Region Conference. Jacksonville, Florida, Robert Meyer Hotel.

Oct. 5-8—Northeast Region Conference. Lord Baltimore Hotel, Baltimore, Md.

Oct. 12-15—South Central Region Conference. Denver, Colo. Cosmopolitan Hotel.

Oct. 20-22—North Central Region Conference, Cleveland, Statler Hilton Hotel.

1960

February—Canadian Region Western Division. Vancouver.

March 14-18—16th Annual Conference and 1960 Corrosion Show. Dallas, Texas, Memorial Auditorium.

Oct. 11-14—Northeast Region Conference. Huntington, W. Va.

Oct. 19-20—North Central Region Conference. Milwaukee.

Oct. 25-28—South Central Region Conference, Mayo Hotel, Tulsa.

1961

March 13-17—17th Annual Conference and 1961 Corrosion Show, Buffalo, N. Y., Hotel Statler.

Oct. 9-11—North Central Region Conference, St. Louis, Chase Park Plaza Hotel.

Oct. 24-27—South Central Region Conference, Houston, Shamrock Hotel.

Oct. 30-Nov. 2—Northeast Region Conference, New York City, Hotel Statler.

SHORT COURSES

1959

September 22-23—9th Annual Shreveport Section Short Course. Centenary College.

November 16-20—4th Annual General Florida Conference 1959 Corrosion Short Course. Key Biscayne Hotel, Miami.

December 7-11—University of Illinois Corrosion Control Short Course. Urbana Campus.

1960

April 27-29—Portland Section Corrosion Control Short Course.

Back Issues of CORROSION

Back issue copies of CORROSION are needed as indicated below:

1947—Nos. 1, 3, 6, 7, 9, 11, 12
1948—Nos. 2, 5, 6, 8, 12
1949—Nos. 1, 2, 3, 7, 9, 10

Any persons who have these issues and want to dispose of them are asked to write to M. A. Glaser, Midland Industrial Finishes Co., East Water St., Waukegan, Ill. who needs them to complete a library collection.

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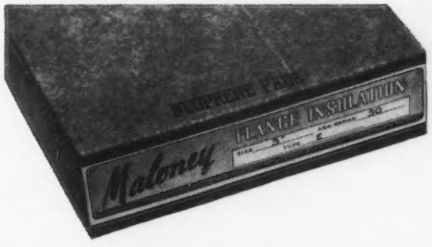
Maloney Flange Insulation is sold in complete sets. A central gasket, bolt sleeves, and insulating washers of "SUPER" Bakelite, with steel washers, make up the package. Three types of central gaskets are available. Type 'D' is an oval, glass reinforced, molded central gasket for ring joint flanges. Type 'E' is a full-face central gasket, with bolt holes, for raised face flanges. Type 'F' is for raised face flanges with an O.D. allowing centering within the bolt circle. Types 'E' and 'F' are available with Neoprene facing, eliminating additional soft gasketing for sealing purposes. All Types are designed to operate at the ASA rating of the flange on which installed.



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'60 Portland Short Course Planned for April 27-29

Preliminary plans are being made for a corrosion control short course to be held April 27-29, 1960, sponsored by the Portland Section.

The course is being developed to relate corrosion control to industries in the Portland area such as pulp and paper, chemicals, communications, transportation and utilties. Questionnaires have been sent to industry representatives to help develop the program.

Plans include an opening discussion on corrosion theory and manifestations of corrosion in its various forms, followed by discussions on corrective measures.

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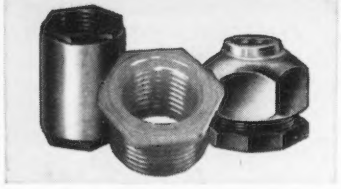

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Western Region News

Seven Symposia Set For Sept. 29-Oct. 1 Regional Conference

Seven technical symposia and an open forum on general corrosion problems have been scheduled for the Sept. 29-Oct. 1 Western Region Conference to be held at the Bakersfield Inn, Bakersfield, Cal.

The symposia are General Corrosion, Petroleum Producing, Utilities, Pipelines, Petroleum Processing, Marine and Chemical Processing.

A list of the papers scheduled to-date are given below with the symposium chairman listed.

Chemical Processing Symposium

Chairman H. F. Keller, Jr., of Jones & Keller, Inc.

Mitigation of Corrosion by Use of Nickel Alloys, by E. G. Holmberg, International Nickel Co.

Precision Coatings for Corrosion Control, by Ira Bechtol, consulting engineer.

General Corrosion Symposium

Chairman E. A. LeDuc, Dearborn Chemical Co.

Isophthalic Polyester Coatings for Protection of Refinery Equipment, by Gorden Johnson, California Research Corp.

Petroleum Producing Symposium

Chairman Bruce Grover, Western Gulf Oil Co.

Cathodic Protection Applied to Internal Surface of Vessels, by R. L. Davis, Superior Oil Co.

An Analysis of Oil Field Corrosion Problems, by J. F. Chittum, California Research Corp.

Field Problems Encountered With Oil Field Scale and Corrosion, by G. Kozlowski, Jones & Keller, Inc.

Bacterial Factors of Corrosion in Oil Producing Operations, to be discussed by a panel.

Cathodic Protection of Oil Well Casings, to be discussed by a panel.

Utilities Symposium

Chairman R. O. Dean, Pacific Gas & Electric Co.

Corrosion Control in Water Treatment Plants, by F. O. Waters, San Diego Water Department.

Cathodic Protection Coordination and Practice, by David T. Jones, Pacific Telephone & Telegraph Co.

A Miscellany of Mitigated Corrosion Problems of a Public Utility Company, by H. H. de Laneux and H. A. Medlock, Pacific Gas and Electric Co.

Pipelines Symposium

Chairman: J. A. Bessom, Richfield Oil Corp.

Cathodic Protection Fundamentals, author to be given later.

Groundbed Development, author to be given later.

Pipeline Interference Problems, by J. W. Ritter, Southern California Gas Co.

Petroleum Processing Symposium

Chairman J. B. Armstrong, Standard Oil Company of California.

Correlation of Corrosion in a Crude Distillation Unit With the Chemistry of the Crudes, by R. L. Piehl, Standard Oil Co., of California.

Corrosimeter Plus Statistical Analysis Yield Unique Approach to Corrosion Problems, by John Patrick and G. E. Moller, Union Oil Company of California.

Selection of Materials for Refinery Corrosion Problems, by A. P. Maradudin, Standard Oil Company of Cal.

Corrosion in Cooling Equipment, by Jack Harris, Wright Chemical Co., and W. B. Harrison, Tidewater Oil Co.

Marine Symposium

Chairman P. W. Hill, Signal Oil and Gas Co.

Three papers are being prepared for this symposium. Authors and titles will be given later.

Open Forum

The open forum on general corrosion problems will be moderated by H. J. Keeling, consulting engineer.

R. M. Evans, Standard Oil Company of Cal., Taft, Cal., is general conference chairman. J. W. Wilt, Honolulu Oil Corp., Taft, Cal., is program chairman.

A final program on the conference will be published in the August issue of CORROSION.

Portland Section officers and members travelled 175 miles to Hermiston to help initiate the NACE program in that area. This special introductory meeting was held in response to interest shown in NACE in the Columbia Valley area of eastern Oregon and Washington.

About 32 interested guests representing various industries of the area heard the Portland Section officers explain the history and organization of NACE and benefits available to members. A brief outline of the fundamentals of corrosion and corrosion prevention was given with a question and discussion period following.

Those attending the meeting expressed agreement that a new NACE section should be established in that area.

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Ralph M. Mattison, Portland Gas and Coke Co., has been appointed secretary-treasurer of the Portland Section for the remaining term of the present officers.

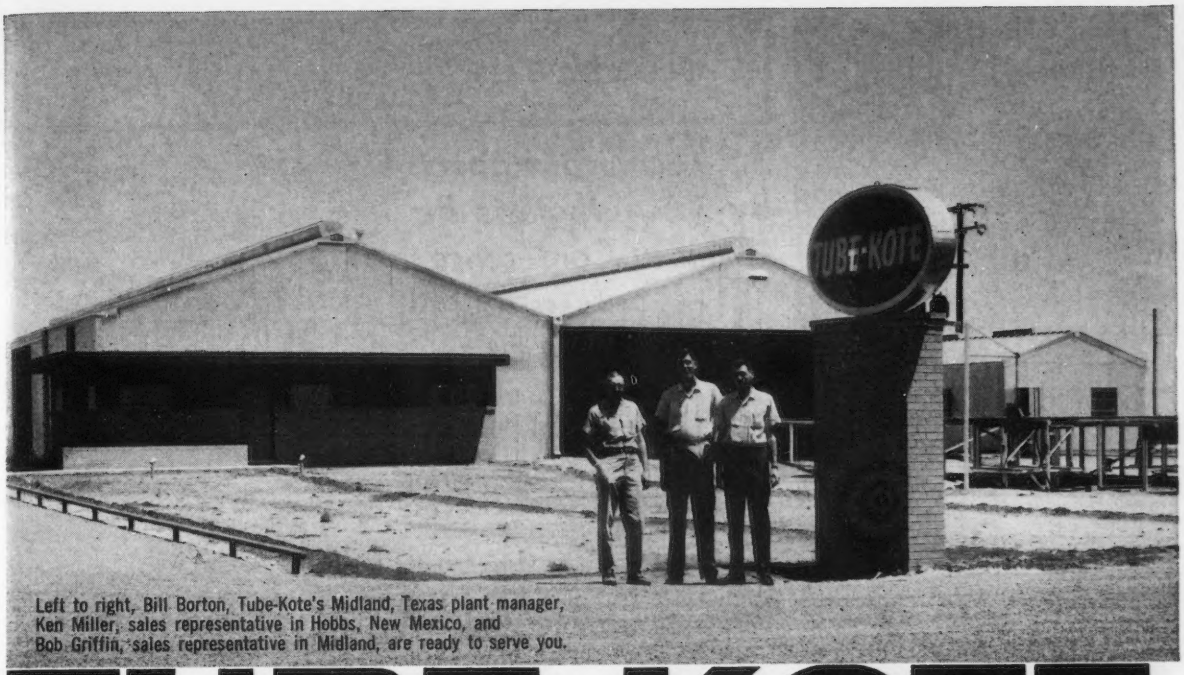
•

San Francisco Bay Area Section had 41 members and guests present at the May 13 meeting. Speaker was Harry E. Thomas, Pacific Gas & Electric Co., Emeryville, Cal., who spoke on the performance of electrical conductors, connectors and splices in marine environments.

•

Los Angeles Section appointed Harry J. Kipps of Signal Oil and Gas Company as vice chairman to replace Bob E. Black, who has been transferred to Houston, Texas.

Speaker at the May 13 meeting was Loren B. Neff of Union Oil Research. He spoke on the control of internal corrosion of tank ships.



Left to right, Bill Borton, Tube-Kote's Midland, Texas plant manager, Ken Miller, sales representative in Hobbs, New Mexico, and Bob Griffin, sales representative in Midland, are ready to serve you.

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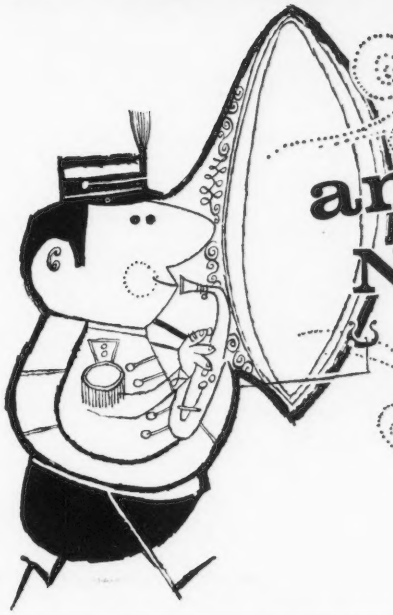

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Northeast Region News

35 Technical Papers By 52 Authors to Be Given at Conference

Thirty-five technical papers by 52 authors will be presented in 8 symposia at the Northeast Region Conference to be held October 5-8 at the Lord Baltimore Hotel in Baltimore, Md.

Industrial plant visits, ladies program, entertainment and technical committee meetings also are planned.

A complete program of the conference will be published in the August issue of CORROSION.

Printed below is a list of the symposia with technical papers to be given in each symposium.

Theories and Principles

Theory of Bimetallic Corrosion by M. J. Pryor.
Theory of Stress Corrosion Cracking by H. R. Copson.
Electrochemistry of Pitting Corrosion by N. D. Greene.

Cathodic Protection—Marine

Service Test Experience With an Automatically Controlled Platinum Anode Cathodic Protection System of an Active Destroyer by L. S. Birnbaum and R. C. Francis.
Electrical Significance of Cathodic Protection on Hazardous Area Steel Docks by J. W. Johnstone and T. A. Mullett.
Some Studies on the Effects of Cathodic Protection on Cavitation Damage by R. L. Burnett, H. S. Preiser and B. H. Tytell.
Effect of Impurity Elements and Water Velocity on Performance of Zinc, Aluminum-Zinc and Magnesium Alloy Anodes by J. L. Basil.

High Temperature Corrosion

Combatting Oil Ash Corrosion in Navy Boilers by B. B. Rosenbaum.
Some Observations on Effect of Caustic Solutions on Corrosion of Steel at 316 C by M. C. Bloom and W. A. Fraser.
Studies of Elevated Temperature Corrosion of Type 310 Stainless Steel by Vanadium Compounds by H. L. Logan.

Protective Coatings—Part 1

Structure and Characteristics of Hot-Dip Galvanized Coatings by H. R. Breslau.
100% Non-Volatile Sprayable Epoxy Coatings by W. C. Naumann.
Coating Performance by Statistical Means by F. E. Cook and A. S. Marthens.
Coal Tar Emulsion Coatings by W. E. Kemp and J. J. Lane.

Corrosion in Handling Water

Chromium Electroplates for Corrosion Protection of Stressed AISI 410 in High Temperature, High Purity Water by H. Suss.
Leaching Action of Various Materials by High Purity Water by J. B. Goodson and E. L. Knoedler.
Power Plant Corrosion by Piero Sturla.
Applications of Aluminum Alloys for Handling Sea Water and Condensate by R. L. Horst.

Protective Coatings—Part 2

Marine Corrosion in Ships by R. S. Capp and B. J. Philibert.
Protection of Ship Hulls Against Corrosion by R. S. Capp and B. J. Philibert.
Roundtable Discussion: Role of Recently Developed Resins in Corrosion Control.
Epoxies by H. H. Flegenheimer.
Use of Polysulphides in coatings by P. W. Atkinson.
Vinyls by R. P. Devoluy.
Urethane Coatings by S. N. Ephraim.
Inorganic Zinc Silicate Coatings by W. A. Guerry.
Role of the New Resins as Linings of Petroleum Fuel Storage Tanks by J. E. Cowling.

Special Corrosion Topics—Part 1

Effect of Composition and Heat Treatment on Stress Corrosion Cracking of Stainless Steel by E. E. Denhard, Jr.
Stress Corrosion Cracking of Austenitic Stainless Steel in Uranyl Sulfate Solution by J. L. English and J. C. Greiss.
Review and Current Status of Bacterial Corrosion by R. L. Starkey.
Interference Aspects of Forced Drainage Installations Designed to Mitigate Corrosion of Underground Cables and Pipes by S. C. Miller.

Corrosion Inhibitors

Application of Corrosion Inhibitors in the Steel Industry by E. H. Phelps.
Researches on Corrosion and Inhibitors: Reaction Velocity in Corrosion of Iron by Aqueous Hydrogen Sulfide and the Effect of Inhibitors by G. S. Gardner.
Inhibitors for Phosphoric Acid by A. D. Fisher.
General Discussion of the Use of Corrosion Inhibitors in Automotive Applications by L. C. Rowe.

Cathodic Protection—General

Cathodic Protection of Steel-Hydrogen Embrittlement Studies by W. H. Bruckner and K. M. Myles.
Properties and Applications of Platinum Surfaced Anodes by C. E. Tirrell.
Preparation and Properties of a Stable Silver-Silver Chloride Electrode for use in Sea Water by M. H. Ortner.
Design Problems in Applying Permanent Cathodic Protection to Glass-Lined Water Heaters by H. C. Fischer.

Special Corrosion Topics—Part 2

The Pentagon Story of Cathodic Protection by L. A. Major.
Corrosion of a Single Crystal of Aluminum by T. H. Orem.

Pittsburgh Section sponsored a tour of the Duquesne Light Company's Shippingport Atomic Power Plant at Shippingport, Pa., as its May 13 meeting.

Section officers for 1959-60 elected at the April meeting are Chairman R. W. Maier of Gulf Oil Corp., Vice Chairman Grant L. Snair of Allegheny Ludlum Steel Corp., Secretary John B. Vrable of New York State Natural Gas Corp., and Treasurer William L. Mathay of United States Steel Corp.

•

Baltimore-Washington Section held a ladies night meeting on May 13. Three films were shown on uses of stainless steel in the home and uses of chemicals and gases obtained from coke in the making of steel.

•

Kanawha Valley Section heard R. E. Gackenbach of American Cyanamid Co., New York, speak on Solid Plastic Materials of Construction at the May 21 meeting.

•

Philadelphia Section heard its program chairman, Norman D. Groves, corrosion engineer with Carpenter Steel Co., speak on case histories of materials failures in the petroleum refining industry at the May 19 meeting.

Presentation of a Past Chairman's Certificate was made to Walter H. Burton, 1958 chairman of the section.

•

Greater Boston Section elected officers for 1959-60 at the May 13 meeting. They are Chairman Benjamin F. Barnwall, Vice Chairman Malcolm L. Mosher, and Secretary-Treasurer Manson Glover.

W. B. Davis of MIT summarized a study of corrosion control by manometric techniques based on oxygen consumption.

•

Three technical committee meetings are scheduled for the Northeast Region Conference to be held October 5-8 at the Lord Baltimore Hotel, Baltimore, Md.

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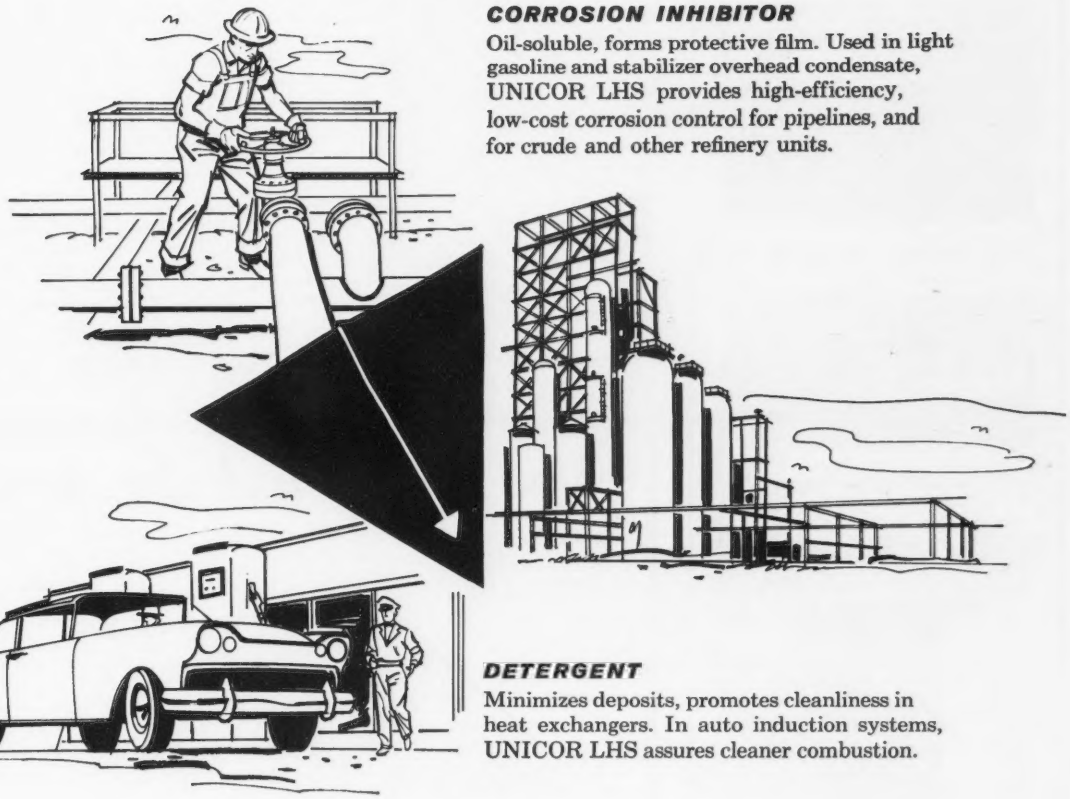

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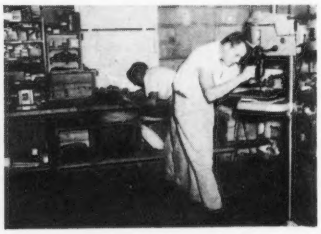

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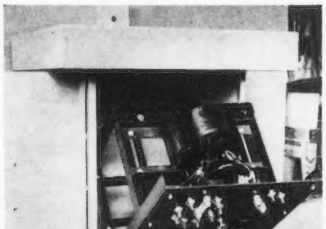

Air Cooled Rectifier

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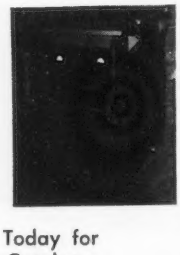

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North Central Region News

North Central Region Conference for 1962 will be held in Detroit rather than Kansas City because the 1962 Annual Conference will be held in the latter city.

•

Greater St. Louis Section heard W. G. Canham of Monsanto Chemical Co., St. Louis, speak on "Should Industrial Painting Theory and Practice Get Together?" at the May 11 meeting.

During the business meeting, chairmen were appointed for the 1961 regional conference to be held at the Chase-Park Plaza Hotel in St. Louis.

•

Eastern Wisconsin Section sponsored a series of six evening lectures from April 20 to May 25 on principles of corrosion and methods of prevention. About 34 registered for the lectures.

Speakers and topics for the series were as follows: Fundamental Principles of Corrosion by L. C. Wasson of A. O. Smith Corp., Coatings for Corrosion Control by H. F. Haase, corrosion consultant, Corrosion Resistant Metals by R. A. Huseby, of A. O. Smith Corp., Inhibitors by F. L. Whitney, Jr., of Monsanto Chemical Co., Cathodic Protection by L. C. Wasson and Design for Corrosion Prevention by Alexander McConnell of George J. Meyer Manufacturing Company.

•

Chicago Section elected new officers for 1959 to serve until January, 1960, when section officers will be elected on a calendar year basis.

Chairman is I. J. Acosta, Crane Company. The other officers are Vice Chairman D. G. Keefe of Swift & Co., Secretary W. H. Schultz of Dearborn Chemical Co., Treasurer D. B. Sheldahl of Sinclair Research Laboratories, Inc., and Trustee H. C. Boone of Peoples Gas Light & Coke Co.

'61 St. Louis Conference Chairmen Are Appointed

Chairmen have been appointed for the 1961 North Central Region Conference to be held October 9-11 at the Chase-Park Plaza Hotel in St. Louis. They are as follows:

General Chairman: Otto Fenner, Monsanto Chemical Company.

Program Chairman: Walter B. Meyer, St. Louis Metallizing Company.

General Arrangements Chairman: J. W. Cushing, Carboline Company.

Entertainment Chairman: Ollie W. Siebert, Monsanto Chemical Company.

About 12 symposia, educational lectures and plant trips of interest to corrosion engineers are being planned as part of the 1961 conference.

•

South Central Region's 1959 Conference will be held October 12-15 at the Cosmopolitan Hotel in Denver, Colo.

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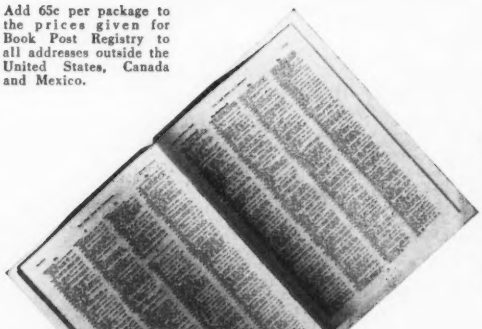

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Foreign Corrosion Control Reports Made for '58

Foreign corrosion study reports made for 1958 has been prepared by the Inter Society Corrosion Committee's subcommittee on Relations with Foreign Organizations. Ten foreign countries are covered: Denmark, France, Germany, Italy, Japan, New Zealand, Norway, Spain and Switzerland.

Correspondents from Austria, Belgium, England, South Africa, Sweden, Australia and the Netherlands did not submit reports.

Addresses of foreign correspondents are given for readers desiring further information.

Members of the sub-committee are P. Delahay, chairman, G. A. Ellinger, F. Fink, Mars G. Fontana, E. Gulbransen, F. L. LaQue, F. N. Speller and H. H. Uhlig.

Denmark

E. Knuth-Winterfeldt, Technical University, Copenhagen.

This report was prepared by Hans Arup, secretary, Corrosion Committee, Danish Academy of Technical Sciences, Oster Voldgade 10, Trappe N, Copenhagen K.

The Corrosion Committee of the Danish Academy of Technical Sciences (ATV), which has a coordinating function between the Danish Corrosion Laboratories, has been active in preparing the Danish participation in the 2nd Scandinavian Corrosion Symposium, which was organized by the Swedish Corrosion Committee in Stockholm May 7-8, 1958. The Danish papers were on a theory for the corrosion and passivation of iron, use of cathodic protection in Denmark and on the role of microorganisms by the corrosion of lead sheathed cables in soil. The last paper has been published in English in Ingenioren, International Edition (2) No. 3, August 1958; G. Bonde and Borge Lunn, "Micro-Organisms and Corrosion of Lead-Sheathed Cables." H. L. Halstrom presented a paper entitled "Is the Phenol Corrosion Theory False?"

A question of the value of some water conditioners with no apparent physical explanation of their advertised function has been discussed in detail, and the Corrosion Committee has made an official statement on the probable effect of one such conditioner.

Work done by the laboratories or committees represented in the Corrosion Committee can be mentioned:

A new committee investigates the corrosion of brass condenser tubes. Transgranular circumferential cracks, normally regarded as fatigue cracks, have been reproduced in the laboratory as a result of static stress corrosion. The effect of cathodic protection (and protector blocks) on the development of these cracks is to be studied.

A committee which has studied the corrosion of oil fired boilers has almost concluded its work, and a final report has been published. The Chemical Department of the Technical University has continued its program of fundamental research on inhibitors. A well equipped laboratory for potentiostatic measurements and short-pulse techniques is now in use. Further information can be obtained from the secretary of the Corrosion Committee, ATV, Technical University of Denmark, Ostervolgade 10, Copenhagen K.

France

G. Chaudron, Faculty of Sciences, University of Paris, 11 rue Pierre Curie, Paris.

The most important investigations are (a) Chemical properties of high-purity metals (Mr. Chaudron, Vitry Laboratories of the Centre National de la Recherche Scientifique). (b) Significant work on iron oxidation at high temperatures (Mr. Bernard at the Sorbonne). (c) A series of investigations for the French Atomic Commission (Mr. Herenguel at the Laboratories of the Trefileries and Laminoirs du Havre). (d) Study of hydrogen and hydrogen sulfide corrosion of steel under pressure (Mr. Herzog, Acieries de Pompey). (e) Fragility and corrosion of steel (Mr. Bastien, Ecole Centrale des Arts et Manufacturers).

The large amount of work being done in France is evidenced in that the laboratory of Mr. Chaudron published 9 papers and that of Mr. Herenguel published 48 papers.

Germany

Prof. G. Schikorr, Chemische Landesuntersuchungsanstalt, Keinestrasse 18, Stuttgart N.

(Continued on Page 84)

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Foreign Corrosion—

(Continued From Page 83)

This report is divided in sections dealing with meetings, and especially with activities of various groups.

Second Congress of European Corrosion Association

This congress, held in Frankfurt/Main June 5-7, 1958, was attended by more than 1000 participants. Six plenary lectures and 55 papers were presented.

Society for Corrosion Studies (Arbeitsgemeinschaft Korrosion). This society prepared a program for corrosion research with support of 1,133,150.60 marks from the Ministry of Economy of the German Federal Government. Twenty-two investigations are being supported.

Advisory Group for Sea Vessel Packing (Beratungstelle für seemassige Verpackung). This group organized two expositions and one meeting on packing techniques and corrosion in overseas shipping. A study was made of meteorological conditions during trips to South America and South Africa.

German Commission for Steel Construction (Deutsche Ausschuss für Stahlban). This commission reported on research on the protection of steel structures by zinc coating and passivity and on sand blasting of steel.

German Bunsen Society for Physical Chemistry (Deutsche Bunsengesellschaft für physikalische Chemie). Twelve papers on the fundamentals of corrosion were presented at the 57th annual meeting of the society. A three-day discussion with 14 papers on metal-gas reactions was also organized.

German Society for Chemical Equipment Manufacturers (Deutsche Gesellschaft für Chemisches Apparatwesen). The following sections of the commission on surface protection were active: non-galvanic metallic coatings, galvanic coatings, non-metallic inorganic coatings, enamels, ceramic and putty coatings and inhibitors.

German Metallurgical Society (Deutsche Gesellschaft für Metallkunde). A symposium on cathodic protection was organized by the corrosion section of this society.

German Bureau of Standards, Section of Plastics (Deutsche Normenausschuss, Fachnormenausschuss Kunststoffe). Specifications were drawn up for the testing of corrosion of copper by paper insulating materials with applied electrical field.

German Bureau of Standards, Section of Material Testing (Deutsche Normenausschuss, Fachnormenausschuss Materialprufung). Specifications and a project for further work were drawn up for the following tests: resistance to impact of enamels, evaluation of non-corroding papers; evaluation of chemical corrosion of steels and zinc by cold and warm water; nickel plating baths; the hull-cell; tin plating and changing humid climates. A glossary of corrosion terms was prepared in German, English, French and Russian. A map of the world climates is also being prepared. Other projects being carried out are specifications for spraying with or without sulfur dioxide atmosphere, non-corroding papers for silver, copper and aluminum.

Society for Research on Combustion Engines (Forschungsvereinigung Verbrennungskraftmaschinen). Research was begun on corrosion and cavitation in cooling circulation systems.

German Chemical Society (Gesellschaft Deutscher Chemiker). The following activities are listed: five public conferences were held by the technical group on paints and pigments; support of investigation on the autooxidation of unsaturated fatty acids; electrical and wetting properties of pigments; mechanical properties, air-permeability, water-permeability, weather resistance and covering properties of paints.

The commission on food and forensic chemistry studied the new food laws concerning the level of metallic impurities resulting from the corrosion of metallic cans. The commission on water chemistry started a study of specifications for phosphate protection of containers and pipes. This commission is also investigating the protection of boilers by hydrazin.

Technical Society for Harbors Construction (Hafenbautechnische Gesellschaft). This group began work on specifications for the evaluation of corrosion by flowing waters.

Technical Society for Ship Building (Schiffbautechnische Gesellschaft). Sessions on the following topics were held: cathodic protection, pitting in tankers, removal of corrosion products from ships hulls and significance of tarnishing in the painting of ship hulls. Specifications were drawn up for painting ships.

Mining Society (Steinkohlenbergbauverein). Cooperative tests were conducted for the non-destructive testing of zinc plating on mining wagons.

German Metallurgical Society (Verein Deutscher Eisenhüttenlente). A study was made of the chemical resistance of steels and nickel-chromium steels containing niobium, molybdenum and vanadium. Specifications were drawn up for stainless, heat-resistant and chemical-resistant steels. The sub-commission on corrosion considered corrosion of steel by additive agents for concrete; corrosion in evaporators for sulfite-containing wastes and tarnishing and atmospheric corrosion of steels. A two-day international meeting also was organized for the discussion of iron passivity and steel and aluminum pitting.

German Engineering Society (Verein Deutscher Ingenieure). Specifications were drawn up for corrosion protection in hot water and steam installations.

Italy

R. Piontelli, Politecnico di Milano, 32 Piazza Leonardo da Vinci, Milan.

Systematic work on the electrode behavior of metallic single crystals has been conducted with improved polarization cells and purification of solution in the laboratory of Electrochemistry, Chemical Physics and Metallurgy of the Politecnico of Milan. See R. Piontelli and G. Poli, *Rend Ist Lomb Sc Lett* **(A) 92,** 601 (1958); R. Piontelli, G. Serravalle and G. Poli, *Rend Accad Lincei* (December 1958) and a group of papers in print. Direct experimental evidence on the catalytic influence of anions on the rate of ionic exchange processes, through competitive adsorption on the electrode surfaces by excluding inhibiting particles, has been attained. Detailed structural investigations on the anodic and cathodic behavior of metallic single crystals have been carried out (papers in print). Passivity phenomena for nickel in aqueous, non-aqueous solutions and fused salts have also been investigated at Milan: R. Piontelli et al. *Z Elektrochem,* **62,** 579, 772 (1958).

In the chemical Physics Department of the Modena University, G. Bianchi and co-workers are studying oxygen reduction and the behavior of aluminum and zinc. Fundamental work on inhibitors has been conducted in the Chemical Laboratory of the Ferrara Iniversity L. Cavallaro).

An extended chapter dealing with corrosion (Piontelli, Bianchi, Jacopetti, Cavallaro, De Carli and others is included in the "Enciclopedia del Petrolio" (ENI), now in print Mario Giordani, Rome, editor).

Japan

T. Fujiwara, I.C.I. (Japan) Ltd., Central Bldg., 2-1 Chrome Kyobashi, Chuo-ku, Tokyo.

Corrosion Committee of the Japan Society for Promotion of Science (K. Ishimura, chairman, Y. Yamamoto, vice chairman, H. Shigeno, secretary). The committee

(Continued on Page 85)

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Foreign Corrosion—

(Continued From Page 84)

members, supporting members and associate members are numbering 72, 62 and 416 respectively. Publication of this group include "Corrosion Engineering" (6 issues per year). A three day lecture meeting in March, 1958, included the following subjects. (1) Standardization of the method of cathodic protection; (2) Improvement of anode materials in cathodic protection; (3) Application of cathodic protection to ships; (4) Organic inhibitors and (5) Corrosion test method for austenitic steels.

A two-day corrosion symposium was held in November at which the following papers were presented for discussion:

Corrosion testing by means of measuring a galvanic generated between the metals (Okano, Ota and Kitamura).

Study of amine type corrosion inhibitors with particular reference to the relationship between corrosion inhibitors and catalyst poison (Fujii and Kobayashi).

Corrosion protection under existence of hexameta-phosphate and other metallic ions (Takeuchi).

Corrosion of packing materials (Takahashi).

Corrosion test for condenser inlet tubes used in steam power plants; Test extended to steel, cast iron, alloy cast iron and stainless steel (Sakae and Endo).

Corrosion protection of propeller shaft of ships (Gada, Miyajima, Morita and Takemura).

Criterion of an effective electric potential in cathodic protection (Maeda).

Maximum protective current applicable to each kind of paint (Hoshino).

New method for finding an actual effect of cathodic protection (Yamamoto and Awa).

Distribution of electric potential in electrically protected condenser tubes (Fukutani, Kakehi and Yamamura).

Characteristics of Al alloys used as anode in cathodic protection (Sakano and Toda).

Cathodic protection of ship body by zinc (Shigeno, Imai and Kuranari).

Cathodic protection of ballast tanks in oil tanker (Fukutani, Kakehi and Kimura).

Cathodic protection of condensers in practice (Kawakami and Oya).

Contact corrosion of titanium and titanium alloys with other metals (Takao).

Thermo electric corrosion (Okamoto and Shimodaira).

A new measurement for determination of cathodic and anodic polarization (Hanada).

Electric Corrosion Control Committee (S. Saheki, chairman). The following subcommittees were formed: Sub-committee for Electric Railway, K. Hoshino in charge, to study controls against corrosion of stray current. Sub-committee for Lead Cable Sheaths, N. Kobayashi in charge, to study cause and prevention of underground corrosion of lead cable sheaths. Sub-committee for Underground Pipes, H. Shigeno in charge, to standardize the method of cathodic protection and coating of underground pipes.

Japan Corrosion Engineering Association (S. Inouye, chairman). The committee and a few sub-committees held meetings to standardize the measurement of corrosion of metals and the quality of corrosion preventive oils, vaporizable inhibitors and anti-corrosive packings. Meetings for lecture and a short course were held to popularize the knowledge on corrosion control of iron and steel products.

Electro-chemical Corrosion Preventive Committee (G. Okamoto, chairman). The committee meetings were held three times during 1958 to discuss theoretical aspects of electro-chemical corrosion.

Association of Surface Treatment of Metals (U. Hashimoto, chairman). Meetings for lecture and a short course were held to promote practical knowledge on plating, metalicon and other surface treatments.

Light Alloy Association. Three sub-committees held meetings to study the surface treatment and cathodic protection of light alloys.

Shipbuilders' Research Association. The committee held meetings every other month to discuss the corrosion protection of ships.

Inter Society Corrosion Symposium. A three day joint corrosion symposium was held in June, 1958 to discuss corrosion problems under auspices of Japan Institute of Metals, Japan Chemical Society and Electrochemical Society at which 41 papers were presented for discussion.

New Zealand

F. J. T. Grigg, The Dominion Laboratory, P.O. Box 8018, Govt. Bldgs., Wellington.

Activities in the Department of Scientific and Industrial Research. Long-term underground corrosion tests are being continued. A progress report has been published in *NZJ Sci*, **1**, 52-69, March (1958). Corrosion investigations related to the use of geothermal steam are continuing, with emphasis on investigations of sulfide stress cracking and a quantitative study of the concentration, distribution and diffusion of hydrogen in steels exposed to H_2S solutions at tem-

(Continued on Page 86)

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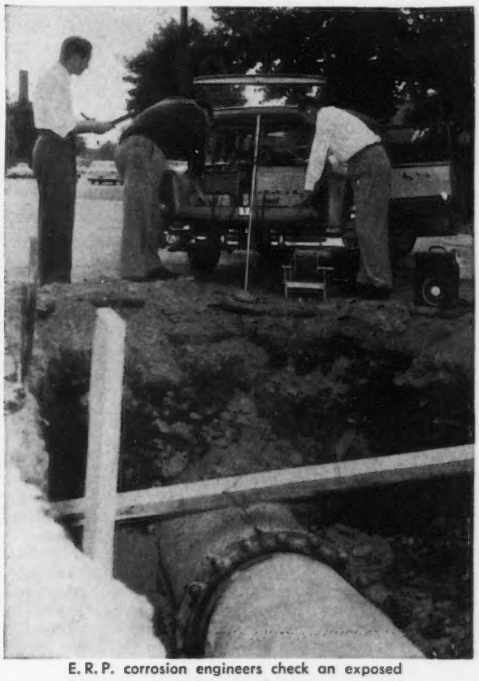

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Foreign Corrosion—

(Continued From Page 85)

peratures to 240 C.

The first New Zealand geothermal steam power plant started operating in 1958. Investigations on the correlation between laboratory and service behavior in the intergranular corrosion of duralumin have been completed; a paper on this subject has been accepted for early publication in the Journal of Applied Chemistry. Studies on crevice corrosion and the galvanic behavior of brazed joints in copper exposed to hot water are continuing.

Activities by other Government Departments. The corrosive effects of preservative chemicals in treated timbers are being investigated by the New Zealand Forest Service.

Commercial Activities. Investigations of corrosion by geothermal steam are being conducted by the Tasman Pulp & Paper Co., Kawerau, New Zealand. No other systematic corrosion investigations by private companies are known to have been conducted in 1958.

Norway

A. B. Winterbottom, Dept. of Metallurgy, Norway Institute of Technology, Trondheim.

This report was prepared by Dr. Bernt Roald, President of the Corrosion Technical Society, Rosenkrantzgt. 7, Oslo.

In later years the activities in the field of corrosion have increased greatly in Norway, and several groups are active in the field, covering fundamental and practical aspects of the problems involved. At the second Scandinavian Corrosion Conference in Stockholm, May 7-9, 1958, eight papers were given covering part of projects that have been concluded recently or are under way.

As an outcome of the increased activity in the field, the Norwegian Association of Corrosion Engineers (Norsk Korrosjonsteknisk Forening) was formed in 1955 as a group of the Polytechnical Society (Den Polytekniske Forening). The association has become a meeting ground for people who have a primary interest in corrosion problems and has sought to bring information on sound corrosion engineering to engineering personnel in need of such knowledge. Last year a well attended course was given on corrosion preventive painting practice, and the lectures and the discussions have been published as a book. Another course on corrosion prevention in fresh water systems, with special regard to boiler corrosion, is underway. Through its membership in the European Corrosion Federation, the association is maintaining contact with other European organizations with an interest in the field.

Spain

E. Jimeno, Ministerio de Marina, Instituto Espagnol de Oceanografia, Madrid.

On October 15, 1958, the Section was constituted for Corrosion and Protection of Materials of the Spanish National Association of Chemists, the statutes having been approved and the Executive Board appointed. The section has 140 affiliated members who work in various industries and research centers. This association is a founder member of the European Federation of Corrosion. In November, 1958, two discussion meetings were held, one dealing with paints of high zinc contents in the protection of metallic structures (Rodriguez Arias) and the other on wood preservation problems (Sanchiz Batalla). The aim of the Section is to group all those interested in corrosion problems who are working independently. To this end, the section publishes a monthly information circular.

Manuel Serra of the department of non-ferrous metals in the May-June issue of Revista de Ciencia Aplicada (Applied Science Review), has published experimental considerations on a new method of metallic protection. This author explains the performances of a semi-industrial plant, of this invention, for the lining with aluminum in a continuous process, of iron wire and by immersion in a bath of molten metal. For every composition of the bath he found a critical temperature of formation of the lining which is exactly the one corresponding to the minimum thickness of the alloy coating. He gives a composition of the bath with which both a better appearance and better mechanical properties are obtained.

In the work of Lamberto A. Rubio on Chemical Nickel Coatings (*Review of the Iron and Steel Institute,* July-September 1958) the influence is studied of the ratio $[Ni^{++}] : [PO_2H_2^-]$, the pH and the temperature in the formation of the coating. The most appropriate values are given. Intensification of the reducing power of hypophosphite is not checked in the presence of the various organic acids tested.

In Bulletin 91, 1958, *Spanish Institute for Oceanography,* a work has been published on direct phosphatation in marine structures by A. Arevalo. Also E. Jimeno and A. Arevalo have published a work in Corrosion et Anticorrosion (February, 1958) entitled "Variations of density of the phosphatic coatings on iron and their protecting value according the time taken to form them."

As a consequence of the foregoing studies, the Spanish Institute of Oceanography decided to bring the protection tests to the direct experimental stage. A series of steel plates were submerged in the Vigo River Estuary sea-water. These test plates remained under water for eight months.

It has been proved that the chlororubber paints are better than the usual ones of linseed oil basis. As regards the previous treatments of the steel plates, a remarkable improvement has been noticed when compared with those not treated, applying phosphoric acid 5M with a brush to some of the testing plates and to others a phosphating solution, by direct brush application of 42 normal points of total acidity and 13.2 normal points of free acidity. Other test plates also were coated with phosphate by immersion in a real phosphating solution to 95 C. Results obtained, though fair, were not as good as those in the two former cases.

These tests are tentative; therefore the fact that the true phosphatization has not yielded better results may be due to some unpredictable circumstance such as unsuitable grain size, etc.

Switzerland

E. Brandenberger, Eidg. Technische Hochschule, Zurich.

The following papers were published:

K. Fobli and A. Brunold. Problems of Lead Cable Corrosion. Part 8—Determination of Nitrate Contents in Soil Water. *Tech Mitteilungen PTT,* 36 (1958).

R. Scheidegger. X-ray Study of Fine Structure of Corrosion Products. *Brown Boveri Mitt,* **45,** 561 (1958).

W. Feitknecht. Diagnosis of Corrosion Causes and Corrosion Products With the Electron Microscope. *Werkstund Korr,* **9,** 556 (1958).

A. Bukowiecki. Corrosion of Steel Cables. *Schqeiz Bau-Ztg,* **76,** 441 (1958).

A. Bukowiecki. Study of Corrosive Properties of Water Containing Oils With Special Consideration of Lubrication and Metal Protection. *Schqeiz Arch, Angew Wiss Tech,* **24,** 20, 263, 295 (1958).

A. Bukowiecki. Investigations of Metal Attack by Cleaning Agents, *Ibid.,* **24,** 355 (1958).

H. Kunzler. Cathodic Protection With Special Power Supplies. *Monatsbull Schqeiz Gas-und Wasserfachm,* **38,** 245 (1958).

C. Tschappat. Investigation of Corrosion Under Pressure and Protection Methods. *Werkst und Korr,* **9,** 613 (1958).

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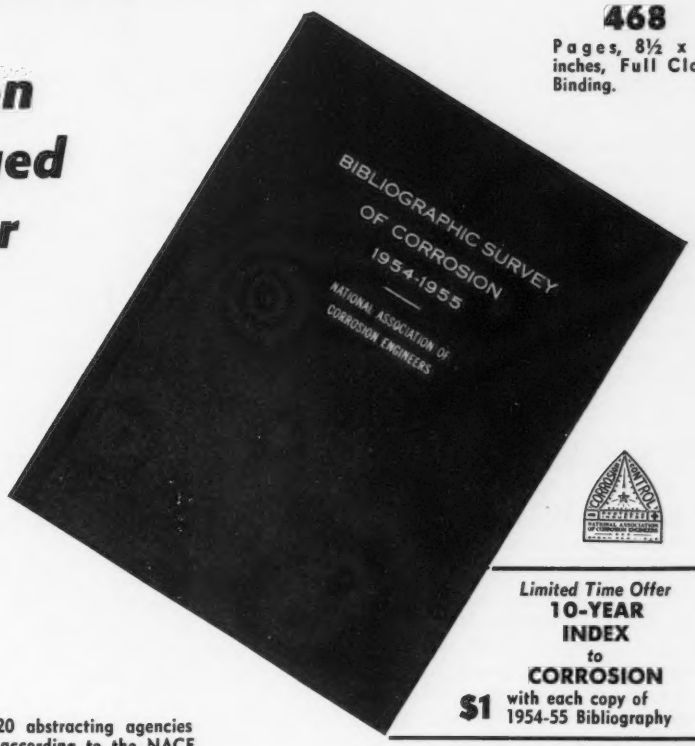

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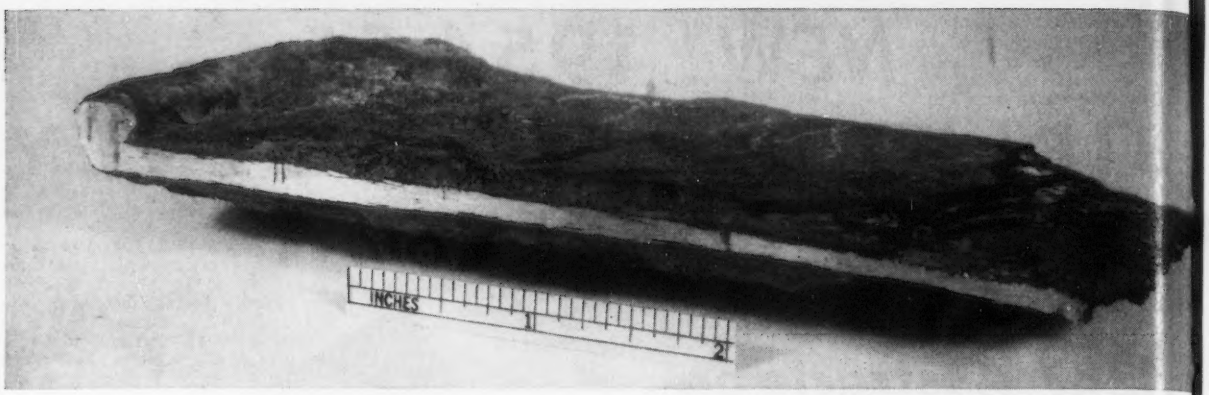
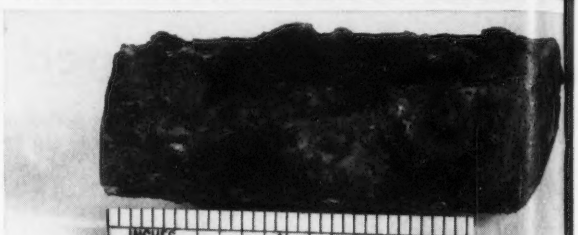


Wrought Iron plate cut by hand from fire curtain of Marina Yacht Harbor Pier, San Francisco, California—The dense and compact nature of Wrought Iron's protective scale is clearly evident. Its adherence is permanent: on plate after 26 years, and on tubular goods after 73 years of salt water service.



Steel tie rod after cleaning and measuring adjacent to deck where paint was still intact—Note chipped and spalled appearance of scale on rimmed steel tie rod. This tie rod was originally 1" material. When cleaned of rust, the rod measured 0.481", 0.490" and 0.697" on three different diameters.

73 years' exposure to salty sea water produced only 3 mils/year maximum pit depth on Wrought Iron

A structural inspection of the Marina Yacht Harbor Piers in San Francisco, Calif., revealed once again, the decided superiority of Wrought Iron over steel in salt water environment.

Maximum pit depth on Wrought Iron after 73 years' exposure to sea water and sea air was 210 mils: approximately 3 mils/year.

Steel similarly located, showed perforation of ¼" plate after 18 years: approximately 14 mils/year. Severance of one-inch steel rods occurred in 26 years.

No case of Wrought Iron perforation was evident. Either in ¼" plate over a period of 26 years, or in tubular goods over a period of 73 years.

Corrosion researchers reported the following detailed findings in their structural inspection:

- Scale on Wrought Iron was heavy and dense. Scale adhered so tightly that a diamond pattern hammer was required to remove it. Scale on steel was equally thick. But it was so friable that light hammering was sufficient to remove it.
- Some scabbing occurred on Wrought Iron at mean high tide (MHT). But, in every case where a scab was hammered off, a dry inactive pit was found.
- At MHT, the maximum pit depth of Wrought Iron was 210 mils. Steel at the same position showed depths of as much as 360 mils. This occurred even though the steel had been in service less than 26 years. Wrought Iron had already withstood 73 years' service.
- In the area of MHT, where the maximum Wrought Iron pit depth was 210 mils in 73 years, the ¼" (250 mils) steel plate had perforated in less than 18 years.
- Wrought Iron, ¼" thick, adjacent to the ¼" steel plate which had perforated in 18 years, showed a maximum loss of 187 mils in 26 years. This occurred in spite of the fact that the Wrought Iron had been subjected to extreme crevice corrosion not obtaining on the steel.
- One-inch steel tie rods passing through MHT showed reduction in diameter ranging from 293 mils to one inch. This would give pit depths (corrosion from one side only) of from 147 to 500 mils in 26 years. This compares with Wrought Iron pits of 210 mils in 73 years.
- No plausible arguments involving small amounts of copper and nickel can be used to invalidate these data. The compositions of the steel and Wrought Iron are very similar for these elements.

The most casual observation reveals use of two different materials in these docks. The Wrought Iron, after 73 years' exposure to salt water and salt air, is today in much better condition than the steel installed 26 years ago.

A complete inspection report of the Marina Yacht Harbor Piers is available on request. Write A. M. Byers Company, Clark Building, Pittsburgh 22, Pa.

BYERS 4-D WROUGHT IRON

TUBULAR AND FLAT ROLLED PRODUCTS

Corrosion costs you more than Wrought Iron

GENERAL NEWS

BOOK NEWS

Field Applied Paints and Coatings. 142 pages, 8½ x 11, hard paper. Publication 563. March, 1959. Building Research Institute, Div. Eng. & Ind. Res., National Academy of Sciences, National Research Council, 2101 Constitution Ave., Washington 25, D. C. Per Copy, $5.

Papers and discussions presented at a conference conducted December 3-4, 1958 at the Shoreham Hotel, Washington, D. C. Discussions of generalized nature on coatings for interiors and exteriors of buildings, special paints, color, economics and future of paints and coatings. Paints for all kinds of wood, masonry and metal services are considered.

Symposium on Basic Mechanisms of Fatigue. 121 pages, 6 x 9 inches, hard paper cover. ASTM Spec. Pub. 237. January, 1959. American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa. Per copy, $3.75.

Papers and discussions presented at the 61st annual ASTM meeting in Boston, Mass. June 23, 1958. Contents include. Progress Report on Dislocation Behavior in Lithium Fluoride Crystals during Cyclic Loading, Fatigue Crack Formation in Silver Chloride; Internal Friction, Plastic Strain and Fatigue in Metals and Semiconductors; Cycle-Dependent Stress Relaxation and Recent Observations on Fatigue in Metals.

3-Year Interim Report of Corrosion Tests on Metallized Coatings. 8 pages 8½ x 11, paper. 1958. American Welding Society, 33 West 39th St., New York 18, N. Y. Availability not given.

Gives information in narrative and tables of corrosion tests on metallized coatings prepared by AWS Commitee on Metallizing. Lists eight test sites used and illustrates test racks and specimen used in the exposures.

Minutes of the Seventh Annual Atomic Energy Commission Welding Conference. 253 pages, 8½ x 11, paper, 1959. U. S. Atomic Energy Commission, Technical Information Service Extension, Oak Ridge, Tenn. Available from Office of Technical Services, Department of Commerce, Washington 25, D. C. Price per copy $2.50

Part 1 presents minutes of joint AEC-ASM Welding Forum World Metallurgical Congress held November 6-8, 1958, in Chicago. Part 2 presents results of the AEC Welding Committee meeting.

Manhole Air Conditioning

Air conditioning in manholes during cable splicing is essential, according to a report in the Industrial Research Newsletter published by Armour Research Foundation. Both humidity and temperature must be controlled to avoid breakdown and contamination of tape insulation.

NACE Members Give Papers At Illinois Symposium

Four NACE members presented papers at the Corrosion Inhibition Symposium held May 7 at Illinois Institute of Technology. They were Norman Hackerman, University of Texas, Austin, Howard T. Francis, Armour Research Foundation, Chicago, W. Roger Buck, III, Virginia Institute for Scientific Research, Richmond, and Milton Stern, Union Carbide Metals Co., Niagara Falls, N. Y.

Papers presented included Molecular Structure and Corrosion Inhibition, Role of Film Formation in Corrosion Inhibition, Mapping Inhibitor Action, Metallic Cations as Corrosion Inhibitors and Corrosion Activators, Observations on the Mechanism of Passivating-Type Inhibitors, and Electrochemical and Electrostatic Factors in Inhibition.

Water Samples Analyzed By New Automatic Machine

Water samples are being analyzed at the rate of 60 per hour by a machine called the Autoanalyzer. It is a photoelectric colorimeter equipped with automatic devices for continuous sampling, addition of reagents, separation of impurities, mixing, measuring of color and recording of results.

Manufactured by Technicon Controls, Inc., Chauncey, N. Y., the machine can measure materials down to parts per billion in water.

Technical Writing Course Offered by Correspondence

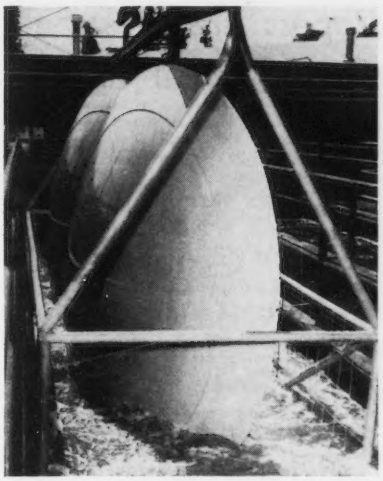
A new correspondence course in scientific and technical writing is being offered by the University of California Extension Service. The course is taught by Hardy Hoover, technical editor for Atomics International.

Further information and application for enrollment can be obtained from Department of Correspondence Instruction, University Extension, University of California, Berkeley 4, Cal.

Metallurgy Meeting Dates Set for Nov. 2-5, Chicago

A comprehensive program in the fields of physical and extractive metallurgy is planned by the Metallurgical Society of AIME for its fall meeting, November 2-5, in the Morrison Hotel, Chicago.

Topics to be included are chemistry and physics of metals, preparation of refractory metals and their properties, extractive metallurgy of refractory metals and their properties, extractive metallurgy of refractory metals and refining of pure metals.



CHEMICAL MILLING PROCESS is being used to mill close tolerances on large spun parts as the bulkhead for a missile shown above.

Chemical Milling Achieves Precise Removal of Metal

New techniques for precise removal of excess metal by chemical milling instead of conventional machining were demonstrated to representatives of the Armed Forces Industrial College by United States Chemical Milling Corp., Manhattan Beach, Cal. Chemical milling was demonstrated as one of the more economical means of machining the exotic metals used in supersonic aircraft and missiles.

Tubing Technical Handbook Revised Edition Available

Seamless aircraft tubing, carbon and alloy steels are described in a revised edition of Ohio Seamless Tube's Technical Handbook A-2. It includes definitions, military and AMS specifications, sizes, tolerances, sampling, testing, packing, marking, machining, heat treating, properties and mill practices. Copies are available from Ohio Seamless Tube Division of Copperweld Steel Co., Shelby, Ohio.

NEW PERIODICALS

The Indian & Eastern Engineer. (In English) Published by M. P. N. de Souza, Sohrab House, 235, Dr. D. Naoroji Road, Bombay-1, India. Price per single copy, Rs. 2.00.

The February, 1959, issue noted that the magazine was celebrating its 101st year of publication. The index page states the magazine is the oldest and foremost engineering journal in India. Contains articles on many aspects of engineering from road construction, atomic energy and rocketry to technical education and engineering colleges.

USSR Corrosion Congress Papers and Authors

About 200 Technical Papers Are Given On Corrosion Topics

Titles and authors of papers presented at the May, 1958, Union of Soviet Socialist Republics Corrosion Congress are given below:

PLENARY SESSION LECTURES

Electrochemical Kinetics and the Theory of Corrosion, by A. N. Frumkin.

Development of an Electrochemical Theory of Corrosion of Metals, by N. D. Tomashov.

Destruction of Metals by the Joint Action of Corrosive and Mechanical Factors, by S. G. Vedenkin.

Theory of Atmospheric Corrosion, by I. L. Rosenfel'd.

Development of a Theory of Corrosion and Protection of Metals in Oxidising Media, by V. P. Batrakov.

Inhibition of Corrosion of Metals, by S. A. Balezin.

Struggle against Corrosion by means of Non-Metallic Chemically Resistant Materials, by I. Ya.Klinov.

Modern Developments in Electrodeposited Coatings, by A. T. Vagramyan and N. T. Kydryavst.

Corrosion of Steel in High-Temperature Water, by E. C. Potter

SECTION 1—SPECIALIST SECTIONS

General Corrosion

1. General Problems of Metallic Corrosion

On the Mechanism of Corrosion of Metals by Oxygen Depolarization, by A. I. Krasilshchikov, et al.

Problems of Metallic Corrosion Influenced by Oxygen Depolarization, by N. P. Zhuk.

On the Mechanism of Inhibition and Acceleration of Corrosion of Iron in Aqueous Solutions of Salt Mixtures, by L. K. Lepin, et al.

Anodic Behavior of Magnesium, by V. S. Komissarova and A. I. Golubev.

Investigation of the Electrochemical Behavior of L & B Brasses, by V. V. Skorchelletti, et al.

Mechanism of Anodic Passivation of Metals and Alloys in Electrolytes, by Ya.M. Kolotyrkin and N.Ya. Bune, et al.

Influence of Ionization on the Corrosion of Metals, by A. V. Byalobsheskii.

Corrosive and Electrochemical Behavior of Stainless Steel in Active State in Solutions of Nitric Acid, by F. N. Mirolubov, M. M. Kurtepov, and N. D. Tomashov.

Influence of Composition and Heat Treatment of Steel and also the Effect of Composition of the Corrosive Medium on the Inter Crystalline Corrosion of Steel Type K18N9, by N. D. Tomashov, et al.

Corrosion Resistance of Aluminum Magnesium Alloys, by V. N. Jordanskii.

Corrosion Protection of High Strength Alluminium Alloys, by S. E. Pavlov, et al.

Investigation of the Structure of Protective Films on Metals by Adsorptimetric Methods, by V. S. Nabokov, N. O. Tomashov, et al.

Oxygen Electrode at Iron, by A. S. Afanas'ev, et al.

2. Corrosion of Metals under Stress

Corrosion Cracking of Austenitic Steels in Alkaline Media at Elevated Temperatures, by A. V. Ryabchenkov, et al.

Influence of Constructional Factors on Resistance to Corrosion Fatigue, by L. A. Glikman.

Increase of Corrosion Resistance of Pump Piston Rods by the Use of Surface Hardening, by E. P. Fedorova, et al.

Corrosion Resistance of Steel in the Water Strata of Petroleum Deposits, by I. L. Faerman.

Investigation of Fretting Corrosion of Metals, by O. N. Myravkin and A. V. Ryabchenkov.

Influence of Fretting Corrosion on Total Fatigue Resistance, by V. A. Veller.

Cavitation Destruction of Bearings Lined with Babbitt Metal, by N. A. Bushe.

Influence of Cathodic and Anodic Polarization on the Velocity of Corrosion Cracking of Steel 1x18N9, by V. V. Romanov, et al.

Cavitation and Corrosion Resistance of Different Steels and Copper Base Alloys, by M. G. Timberulatov, et al.

Corrosion Fatigue Resistance of Aluminum Alloys, by V. S. Sinyavskii and S. G. Vedenkin.

3. Atmospheric Corrosion of Metals

Electrochemical Investigation of the Corrosion of Metals by Moist Atmospheres with Different Amount of Sulphurous Gases, by G. B. Clark and N. D. Tomashov, et al.

Corrosion of Constructional Steel in Natural Conditions, by V. G. Chernashkin, et al.

Atmospheric Corrosion of Steel, by A. A. Bavakov, et al.

4. Corrosion of Metals in Sea Water and Other Neutral Solutions

On the Mechanism of Corrosion of Metals by Periodic Moistening with Electrolytes, by I. L. Rosenfel'd, et al.

A Model System for Corrosion Testing in Moving Sea Water, by B. V. Strokan.

Sea Water Corrosion of Steel and Resistant Alloys for Marine Construction, Ships & Pipe-lines, by V. F. Negreev, et al.

Investigation of the Corrosion Stability of Carbon and Low Alloy Steels in the Barents Sea, by G. C. Koshelev.

Effectiveness of Zinc Protection in Moving Media, by A. I. Zuv.

Metal Anodes for the Cathodic Protection of Steel Structures in Sea Waters, by S. A. Ivanov.

Protective Methods for Zinc and Aluminum Base Alloys Containing Calcium, by A. F. Zhurakhovski, et al.

Investigation of Corrosion of Cupro-nickel Condenser Tubes in Flowing Water, by A. E. Gopius, et al.

Study of the Corrosion Resistance of Different Non-ferrous Alloys in Sea Water and Sea Mist, by E. M. Zimneva.

Prevention of Corrosion of Aluminium Tubes in Car Radiators, by F. B. Zlomyanskaya, et al.

Study of the Corrosion of Metals in Mineral-Containing Waters, by F. N. Tavidz, et al.

5. Gaseous Corrosion of Metals and Corrosion of Metals in Molten Salts

Nature of the Heat Resistance of Chrome Nickel Alloys, by N. I. Tugarinov, et al.

Investigations in the Fields of Gaseous Corrosion and of its Influence on the Heat Resisting Properties of Steel, by A. V. Ryabchenkov, et al.

Corrosion of Steel in Molten Mixtures of Sulphate Salts, by M. M. Cristall', et al.

Cleaning Tubes of High Alloy Steel, by N. V. Boyoyavlenskaya, et al.

Sodium Hydride Descaling, by Z. P. Men'shikova, et al.

6. Corrosion of Titanium and its Alloys

Corrosion Resistance and Electrochemical Properties of Titanium and its Alloys, by V. V. Andreeva, et al.

Influence of Halogen Ions of the Corrosion and Electrochemical Behavior of Titanium in Medium-Strength Acids, by R. M. Al'tovskii and N. D. Tomashov.

Influence of Contact with Different Metals on the Corrosion Resistance of Titanium and its Alloys, by L. Chirkova-Corrosion Resistance of Titanium, by J. B. Cotton, et al.

7. Corrosion of Metals in the Chemical and Other Industries

Corrosion of Metals in Aqueous Solutions of Ammonium and Calcium Nitrate Saturated with Ammonia, by M. L. Rutkovski, et al.

Corrosion of Iron Alloys in Solutions of Sulphuric Acid, by E. V. Zotova.

Alloys for Equipment Operating in Sulphuric Acids, by G. L. Schwartz, et al.

Study of Corrosion of Parts of Electrolysers for Electrolysis of Water, by Z. A. Tkachek.

Corrosion of Steel in Hot Concentrated Solutions of Caustic Soda, by F. A. Orlova.

Corrosion of Metals in Chlorine/chlorate Solutions and Corrosion of Apparatus for the Production of Chlorine Diozide and Sodium Chlorite, by R. E. Romanushkine, et al.

Corrosion of Welded Joints in Aluminium Apparatus in Boiling Concentrated Nitric Acid, by F. B. Slomyanskaya, et al.

Corrosion of a Benzole Column in a Coke-Chemical Plant and Apparatus for the Rectification of Crude Phenol, by A. I. Brodovinch, et al.

Corrosion of Equipment in the Process of Direct Hydrogenation of Ethylene, by V. F. Negreev, et al.

Corrosion of Chromium in Special Conditions, by V. N. Poddubnii, et al.

Protection of Apparatus of Compressed Carbon in the Production of Synthetic Mathanoi at G.S.Kh.K., by Stalinorsk Chemical Combine.

Corrosion and Protection of Equipment in Plant for the Production of Urea at G.S.Kh.K., by Stalinorsk Chemical Combine.

Investigation of Corrosion Resistance of Different Metals in Grape Wines, by F. N. Tavadze, et al.

Corrosion Resistance of Stainless Steel in Digesters Handling Peat Tar Waters, by M. V. Konradi.

SECTION 2

Underground Corrosion

1. General Problems of Underground Corrosion

Investigation of the Electrochemical Processes of Soil Corrosion of Metals, by N. D. Tomashov and Yu. N. Mikhailovskii.

Construction of the Gas Pipe-line Network in the Sixth Five-year Plan and its Protection against Corrosion, by N. I. Ryabtsev.

On Methods of Determining Corrosion Dangers of Soils, and of Corrosive Conditions in Underground Constructions in Field Conditions, by A. A. Spirin, et al.

Corrosion Investigations in the Territory of Moscow City, by L. I. Akinfiev.

Experiments in the Protection of Underground Constructions in Large Cities, by N. A. Tsekun.

The Protection of Urban Underground Gas Pipes, by K. A. Aliev.

2. Anti-Corrosion Coatings for Underground Constructions

Theory and Design of Protective Coatings for Underground Metallic Pipe-lines, by L. Ya.Tsikerman.

Protection from Underground Corrosion of Ferro-Concrete Transport Constructions, by V. S. Artamonov.

Experiments on the use of Polyvinyl Chloride Plastics for Protection of Underground Pipe-lines, by S. R. Rafinkov, et al.

Resistance of Bitumen Insulating Coatings under Conditions of Cathodic Protection, by E. A. Andreeva.

Enamel Coatings for Protection of Underground Pipe-Lines, by M. A. Aliev.

3. Electrochemical Protection of Underground Constructions

On the Possibility of Intensive Corrosion of Lead-Coated Cables by Continuous Applications of Cathodir Polarization Currents and Protective Potentials, by A. F. Marchenko.

On the Influence of Certain Factors on the Distribution of Potentials along a pipe-line during Electro-chemical Protection, by M. S. Trifenl'.

Cathodic Protection of Trunk Pipe-lines and Experiments on its application, by V. G. Kotik.

Distribution of Current and Potential along Equipment during its Cathodic Protection, by V. V. Krasnoyarskii.

Effectiveness of Electro-protection of the Gas Line Dashava-Kiev, by I. N. Frantzevich, et al.

4. Protection of Underground Constructions from Corrosion Effected by Stray Currents

Experiments on the Application of Electro-drainage of the Protection of Gas Pipe-lines in Industrial Regions of the Ukraine, by I. N. Frantzevich, et al.

Electricity Distribution of the Electric Transport System of Towns and its Influence on the Problems of Protecting Structures for Corrosion by Stray Currents, by D. K. Tomylanovich.

Methods of Effective Isolation during Reduction of Leaks from Traction Currents from Rails of Electrified Railways, by I. M. Ershov.

Electroprotection of a Trunk Pipe-line from Corrosion Caused by Stray Currents and the Choice of Means of Protection, by P. G. Dorshenko, et al.

Regulation of Potential at Drainage Points of the Rail Network of Trams with the Help of Special Rectifying Plant, by E. V. Chebotarev.

Electro-corrosion of Ferro-Concrete, by I. A. Cornfel'd, et al.

Influence of Currents from Electric Transmission Lines in the Constant Current Systems "Conductor-earth" on the Corrosion of Underground Metallic Constructions. by K. K. Nickol'skii.

Calculation of the Corrosion Hazard of Covered Cable Bonds Occurring in the Field by Stray Currents of Electricity, by L. D. Razumov.

SECTION 3

Inhibitors of Corrosion and Protective Films

1. Volatile Inhibitors

On the Mechanism of the Protection of Metals from Corrosion by Volatile Inhibitors, by V. P. Persiyantseva and I. L. Rosenfel'd, et al.

Study of Phosphates of Monoethanolamine as Inhibitors of Atmospheric Corrosion, by R. M. Golubeva, et al.

Theoretical Basis and Practical Results of Conservation of Machinery with the Assistance of Other Volatile Inhibitors, by O. I. Golyanitskii.

Tests of Protection by Means of Volatile Inhibitors for Components during Long Term Storage, by Lekareva.

2. Retardation of Corrosion with Neutral, Acid, and Alkaline Inhibitors

On the Joint Action of Sulphur-Containing Inorganic Ions and Organic Inhibitors on the Acidic Corrosion and Brittleness of Iron, by Z. A. Iofa.

Influence of Amines and Some of their Salts on Corrosion of Carbon Steel, by G. V. Kleshcheva and S. A. Balezin.

Joint Influence of Urotropine, etc. and Potassium Iodide on the Corrosion of Iron in Hydrochloric and Sulphuric Acids, by I. P. Anoschchenko.

Investigation of the Products of Corrosion with Low Carbon Steel in Neutral and Weakly Acid Media with Additions of Promoters and Inhibitors, by N. M. Malin.

Passivation of Some Metallic Components under Flow of Electrolytes, by S. A. Balezin, et al.

Organic Inhibitors of Corrosion of Steel in Waters Containing Hydrogen Sulphide, by T. Kh. Monakhova and V. F. Negreev, et al.

Corrosion of Steel in Cement and Appropriate Corrosion Inhibitors, by V. B. Ratinov, et al.

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USSR Corrosion–

(Continued From Page 90)

Combined Protection of Iron from Corrosion in Neutral and Weakly Alkaline Solutions, by V. P. Grigor'ey and L. I. Antropov.

Investigation of Protection from Attack by Mixtures of Inhibitors of Corrosion in Neutral Solutions, by A. S. Afanas'ev, et al.

Influence of Corrosion Inhibitor PB-5 on the Corrosion Cracking of Steel 18/9 in 42% Solutions of MgCl2, by V. V. Romanov and S. A. Balezin, et al.

Corrosion of Copper Alloys in Hydraulic Fluids, by V. N. Poddubnii, et al.

Passivation of Cast Iron and Steel Components by Solutions of Sodium Nitrate to Prevent Corrosion on Long Storage, by I. F. Gridnev and I. L. Rosenfel'd.

Influence of Temperature on Corrosion Resistance and Electrochemical Properties of Low Carbon Steel in Acid Inhibited Media and the Application of Sodium Arsenate as a Corrosion Inhibitor of Steel in Aqueous Solutions of Sulphuric and Hydrochloric Acids, by V. A. Ehitrov, et al.

Mechanism of Protection by Means of Thiourea During the Solution of Steel in Acids, by A. I. Fil'ko and S. A. Balzin.

Inhibition of Corrosion of Lead in Water, by A. R. Myagkova and I. N. Putilova.

Selection of Inhibitors for Protection from Corrosion of Metals under Attack by Explosive Mixtures for Coal Mines, by A. V. Shreider, et al.

3. Inhibition of Corrosion of Hydrocarbons and Protective Greases

Anti-corrosion Additions to Motor Oil, Their Nature and Mechanism of Action, by B. V. Losikov.

Multifunctional Additions to Oils and Greases, Increasing Their Anti-Corrosion Properties, by D. S. Velikovskii, et al.

Investigation of Additions to Oils for the Prevention of Corrosion of Components of Car Engines, by K. S. Ramaiya, et al.

Corrosion Products of Lead in Hydrocarbon Solutions, by I. N. Putilova, et al.

Improvements in Corrosion Inhibitors for Additions to Greases Used for Railway Roller Bearings, by V. G. Kuznetsov, et al.

SECTION 4
Metal Coatings and Chemical Treatment of Metals

1. Degreasing and Pickling of Metals

Chemical and Electrochemical Cleaning of Metals, by P. P. Belyaev, et al.

Technological Processes for Simultaneous Continuous Degreasing and Pickling, by P. A. Galaktionov, et al.

Degreasing Steel in Aqueous Alkaline Solutions, by M. I. Morkhov, et al.

Intensification of Processes for the Degreasing, Pickling, and Cleaning of Metallic Surfaces in an Acoustic Field, by A. A. Baram.

Ultrasonic Degreasing, by V. M. Gevorkyan.

2. Phosphate Coating

On the Mechanism of Formation of Phosphate Coatings on Metals, by V. S. Lapatukhin.

New Oxyphosphate Coatings for Steel Components, by A. F. Krutikov.

Special Properties of Phosphatized Steel in the Presence of Nitrates, by I. I. Khain.

Phosphatization of Chrome Steel and Nonferrous Metals, by M. A. Novitskaya.

Utilization of Ion-Exchange Resins in Chemical and Electrochemical Preparation of Metals, by E. V. Gannishkina, et al.

3. Intensification of Processes of Coating and the Durability of Coatings

On the Mechanism of Electrodeposition of Metals by Currents of Alternating Direction, by A. P. Popkov and A. T. Vagramyan.

Influence of Reversing Currents on the Internal Stresses in Electro-deposited Coatings and on the Distribution of Metal on the Cathode, by G. T. Bakhvalov.

Intensification of the Process for Chrome Plating, by M. A. Shluger, et al.

Chrome-Plating in Self-Regulating Electrolyte, by V. M. Semin.

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CORROSION ENGINEERING DIRECTORY

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USSR Corrosion—

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Electrochemical Chrome-Plating from Solutions of Trivalent Chrome Salts, by I. A. Bodrov and N. T. Kudryavtsev.

Chemical Nickel-Plating, by A. A. Nikiforova and K. M. Gorbunova.

Applications of Chemical Nickel-Plating for Protection from Corrosion and Increasing Wear-Resistance of Components, by S. A. Vishenkov.

Electrolytic Deposition of the Wear-Resistance Compound Nickel-Phosphorus, by M. E. Gol'dstein.

Cathodic Processes During the Electrodeposition of the Alloy Iron-Nickel, by P. S. Titov, et al.

4. Electrolytic Coatings

Electroplating of Light-Alloy Components, by V. I. Lainer.

Deposition of Silver and Cadium from Cyanide-free Electrolytes, by S. Ya. Popov, et al.

Influence of the Structure of Steel on Inclusion of Hydrogen during Electrolytic Process ing, by G. D. Vol'pert.

Zinc Coating of Steel Springs with Conservation of their Mechanical Properties, by A. M. Ginbert, et al.

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Technology of Deposition of Alloys Sn-Zn and Sn-Cd and Their Corrosion Stability in Simulated Tropical Conditions, by P. M. Vyacheslavov.

Selection of Protective Coatings for Components Employed in Tropical Conditions, by F. F. Azhogin, et al.

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Electrolytic Antimonial Coatings, by V. M. Gevorkyan.

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Deep Anodizing of Aluminium Alloy Components, by I. I. Moroz.

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Automatic Electronic Instruments for Maintaining Fixed Current Density, Coating Times and Temperatures of the Electrolytes in Electroplating Baths, by Yu. V. Gorgish-Klushin.

An Electronic Instrument for the Control of Electroplated Coating P.U.R.P.-1., by L. M. Mashevich, et al.

Internal Pressure in Bright Electroplated Deposits, by Yu. V. L'izlov and A. G. Smartsev.

Development of Methods of Analysis of Fluoboric Electrolytes, by E. Lerman.

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Corrosion of External Casings of Underwater Parts of Ships' Hulls and Exploitation of Special Protective Coatings, by I. Ya. Bogorad, et al.

Ethynol Paints and their Utilization in Ship Building, by E. V. Iskra.

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Practical Applications of Polyisobutylene for the Protection from Corrosion of Apparatus and Structures, by D. A. Gofen.

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The Present State and Prospective Production of Glass Tubes in the Soviet Union, by I. E. Shapiro.

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Organization of Anti-Corrosion Service in Chemical Undertakings, by G. Z. Vashin.

Protection from Corrosion in Hydrolysers, Sulphate Spirit, and Glucose Factories, by Verob'eva.

•

NACE's Bibliographic Surveys of Corrosion extend from 1945 to 1955. Prices available upon request.

•

NACE's 17th Annual Conference and 1961 Corrosion Show will be March 13-17, 1961, at the Hotel Statler in Buffalo, N. Y.

POSITIONS WANTED AND AVAILABLE

Active and Junior NACE members and companies seeking salaried employees may run without charge two consecutive advertisements annually under this heading, not over 35 words set in 8 point type. Advertisements to other specifications will be charged for at $10 a column inch.

Positions Available

Sales Engineer: Splendid opportunity for qualified engineer with broad field experience in cathodic protection. Interested in sales development work on relatively new and accepted product. Location in or near Houston but would require extensive traveling principally in Southwest. Preferred age 25-35. Reply guaranteed. Send photo & resume of experience. CORROSION, Box 59-21.

Manufacturers' Agents

Manufacturer's Agent—Major manufacturer of corrosion resistant protective coatings seeking topnotch representatives for exclusive territories of Pittsburgh, Omaha, Louisville, Minneapolis, Knoxville, North and South Carolina. Commission basis. Excellent opportunity in rapidly expanding field. High potential. CORROSION, Box 59-26.

Corrosion control tape coating manufacturer is appointing representatives in various areas. For your opportunity in this growth item submit a complete resume including areas and types of accounts covered to CORROSION, Box 59-24.

Positions Wanted

B. Sc. (Chemistry), M.C.I.C.—married, sales experience industrial chemicals and industrial water corrosion control. Desires employment Western Canada. CORROSION, Box 59-20.

Over Five Years experience in all phases of cathodic protection of pipelines and storage tanks. B. S. Degree. Age 35. Supervising experience. Complete resume on request. CORROSION, Box 59-22.

Patent Law: B. S. in Met. E., LL. B. in Law. Seven years experience in research, development, and production in electrodeposition and corrosion. Member of state bar. Age 28. Married. Desire patent position. Resume on request. CORROSION, Box 59-25.

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Saran Lined Pipe and valves carry hot Fluorides...

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When pipe must carry a constant flow of extremely corrosive mixed fluorides solution . . . when it *must* operate dependably 24 hours a day, seven days a week . . . that's when Saran Lined Pipe is specified by experienced corrosion engineers.

At the world's largest aluminum smelter, in Arvida, Quebec, engineers of the Aluminum Company of Canada, Ltd. (ALCAN), have specified the use of Saran Lined Pipe to handle the flow of mixed hydrogen fluoride and sodium fluoride solution at 100° F.

In this operation, 2000 feet of Saran Lined Pipe and many Saran lined valves are used. Most of the pipe is outdoors, resting on brackets. Because of the high physical strength of Saran Lined Pipe, no damage results from changing temperatures, or from the often-great temperature differential between the pipe's contents and the outside air. Alcan's engineers specified Saran Lined Pipe for this installation because of previous highly successful experience with it in similar installations at Alcan's five other smelters.

Whenever a dependable piping system is required, whatever the degree of corrosion or chemical activity, consider Saran Lined Pipe. Saran Lined Pipe, fittings, valves and pumps are available for systems operating from vacuum to 300 psi, from below zero to 200°F. They can be cut, fitted and modified easily in the field without special equipment. For more information, write Saran Lined Pipe Company, 2415 Burdette Avenue, Ferndale, Michigan, Dept. 2284AU7.

THE DOW CHEMICAL COMPANY • MIDLAND, MICHIGAN

Equipment Services NEW PRODUCTS Materials Literature

Air Cleaners

Electro-Air Cleaner Co., Inc., Olivia and Sproul Streets, McKees Rocks, Pa., has developed a portable electronic air cleaner to handle 225 cubic feet of air per minute and to remove 90 percent of air-borne particles, including those as small as one one-hundredth of a micron.

Alloys

Haynes Stellite Co., Division of Union Carbide Corp., 420 Lexington Ave., New York 17, N. Y., has developed processes for cold-rolling sheets of high temperature alloys with greater dimensions of width and length than formerly possible. Sheets measuring 0.10 inch thick, 36 inches wide and 96 inches long are being rolled.

Aluminum

Aluminum Foil Booklet listing uses of aluminum foil in the building, electrical, packaging, insulating and other industries is available from Aluminum Company of America, 1501 Alcoa Bldg., Pittsburgh 19, Pa.

•

Super Purity Aluminum production is being expanded at Kaiser Aluminum and Chemical Corp., Kaiser Bldg., Oakland 12, Cal. New refining cells to go into production this fall will triple the firm's production of an aluminum that is claimed to be 99.99 percent pure. Industrial uses are as catalysts for producing high octane gasoline, foil for electrical capacitors and decorative uses in automobiles, appliances and costume jewelry.

•

Adaptaloy, a new aluminum alloy has been developed with high elongation and impact strength and the ability to sustain bending and deformation, according to Federated Metals Division of American Smelting and Refining Co., 120 Broadway, New York 5, N. Y.

Ceramics

Magnesia Ceramics, 99.8 percent pure, have been developed by Corning Glass Works, Corning, N. Y. They are suitable for refractory and electrical applications. According to Corning, a uniform, fine-grained structure gives the ceramics a flexural strength of 15,500 psi at room temperature.

•

LV Series Pumps for handling highly corrosive and abrasive materials are described in a brochure published by General Ceramics Corp., Chemical Equipment Division, Keasbey, N. J. Applications and type of ceramic lining used in the pumps are described.

Coatings—Metallic

Drygalv, a zinc-rich liquid to provide a 95 percent metallic zinc film for corrosion control on ferrous metals and welded joints, is described in a bulletin released by American Solder & Flux Co., 19th and Willard St., Philadelphia 40, Pa. Drygalv can be applied by brush or spray with minimum surface preparation.

Coatings—Organic

Coatings Chart entitled "Comparison of Maintenance Coating Systems" has been released by Carboline Co., 32 Hanley Industrial Court, Brentwood 17, Mo. The chart compares 17 standard systems from standpoint of corrosion resistance. Compatibility for coatings when used over old paint, surface preparation and other topics are included.

•

New Wetting Additive for rust-inhibiting red lead paints on steel surfaces is being tested by the Lead Industries Association, New York, N. Y. The association is attempting to develop a vehicle that will wet steel as well as or better than linseed oil without the slow-drying characteristics of linseed oil.

Filters

Porous Metal Filter Discs, constructed of stainless steel fibers, are being produced by Purolator Products, Inc., Rahway, N. J. The discs are designed for operations with wide temperature ranges and differential pressures to 200 psi. They resist most concentrations of nitric acid.

•

Sel-Rex Corporation, Nutley, N. J., has developed an improved model of its Pre-Met portable solution filter which features a rotating magnetic field to drive the pump impeller to eliminate the need for seals or stuffing glands.

Inhibitors

Orchard Paper Co., 3914 N. Union Blvd., St. Louis 15, Mo., has published the Orchard Inhibitor Paper Manual which gives a non-technical explanation of inhibitors and describes the firm's products.

•

Corrosion Inhibitor CS is a new patented compound designed to prevent corrosion of steel, copper, brass, aluminum and solder in closed recirculating water systems. It was tested for four years in various systems. Detailed information is available from Hagan Chemicals & Controls, Inc., Box 1346, Pittsburgh, Pa.

Non-Metallics

Activated Carbon produced from petroleum materials rather than coconut shell charcoal is being produced by National Carbon Company, Division of Union Carbide Corp., 100 East 42nd St., New York 17, N. Y.

•

Aicco-Sol, a new synthetic magnesium silicate, is being produced by American Industrial Chemical Co., division of Amerace Corp., at Butler, N. J. The silicate absorbs moisture, gases, most common color and odor-forming bodies, weak acids and polar compounds. Applications will be in controlling thixotropy of plastic coatings and as catalyst and catalyst carrier.

New Kennametal Compositions are available for corrosion-wear parts made by Kennametal Inc., Latrobe, Pa. They are a binderless carbide of tantalum and tungsten called Grade K601 and a tungsten carbide with chrome-cobalt binder called Grade K701.

•

Impervite Modular Exchanger System, manufactured by Falls Industries, Inc., Aurora Rd., Solon, Ohio, permits use of impervious graphite in highly corrosive applications where true counter-current flow is desired or where highly flexible heat transfer capacity is required.

Plastics

Byers PVC Pipe is being distributed in the Southwest by Southland Supply Co., 2034 Amelia St., Dallas, Texas.

•

Armor-Crete, a chemical and abrasion resistant topping for floors of concrete, wood, steel and other materials, is manufactured by Preco Chemical Corp., Allen Blvd., Farmingdale, N. Y.

•

Fluoroflex-T Transfer Hose, developed by Resistoflex Corp., Roseland, N. J., is an abrasion resistant rubber jacket hose reinforced with spiraled wire. A layer of Fluoroflex-T extends through the hose and over flanges to eliminate contact between fluids and metal flanges.

•

Thred-Tape, a pipe joint sealer made from Du Pont Teflon, is designed to seal threaded connections in pipe handling industrial acids, corrosives, caustics, hydraulic fluids and aromatic fuels. Made by Crane Packing Co., 6400 Oakton St., Morton Grove, Ill., the tape can be used on plastic, aluminum, stainless steel, ceramics, synthetic rubber and carbon pipe.

•

Rome Cable Booklet entitled PVC Insulated Leads for Oil Filled Transformers is available from Rome Cable Corp., Rome, N. Y. It discusses insulation properties required for oil-immersed transformer leads and compares these requirements with properties of Synthinol 901, a PVC compound developed by Rome Cable.

•

Bubble Caps and Risers manufactured from a proprietary thermosetting plastic material are being produced by Wyatt's Plastics, Inc., 5928 Katy, Houston, Texas. The material has been tested for 10 months in a tower contacting 55 to 65 percent sulfuric acid at 340 F.

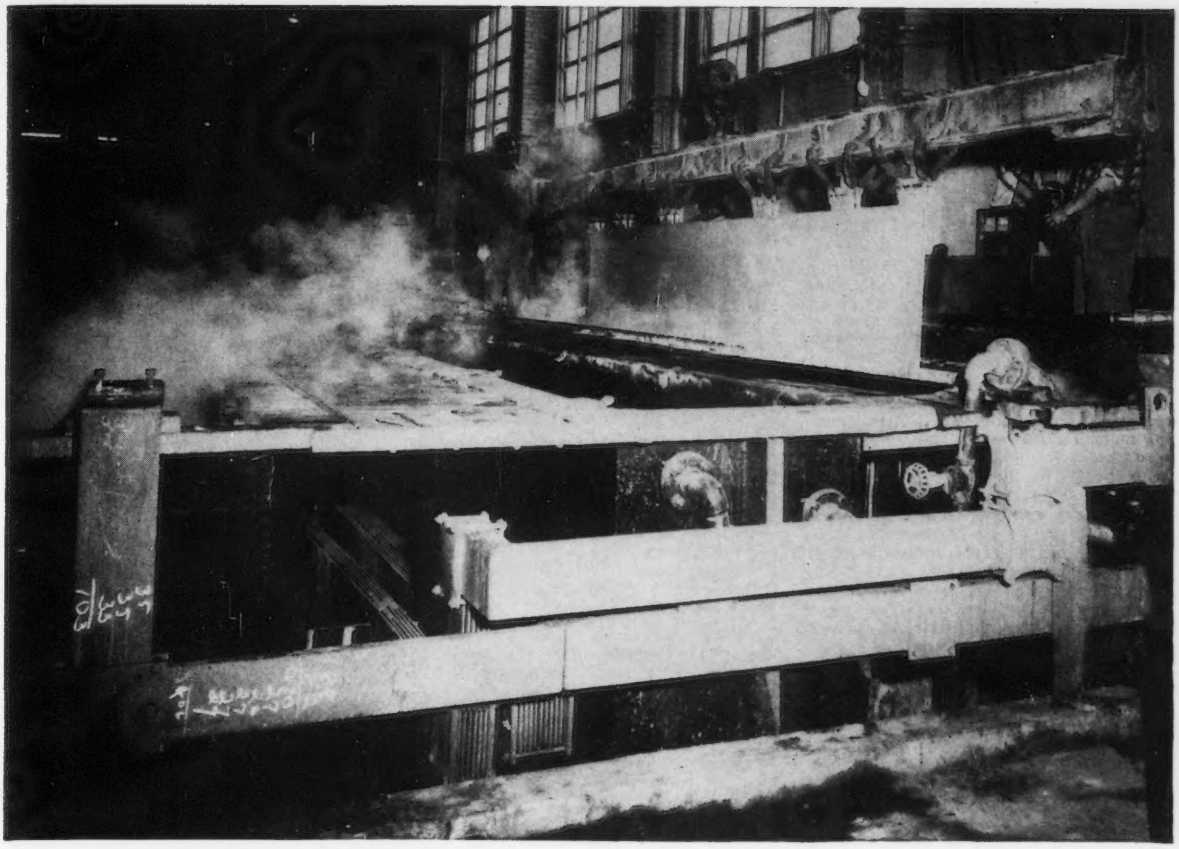
•

Delrin Acetal Resin is being used in the production of tiny injection molded plastic parts by Gries Reproducer Corp., New Rochelle, N. Y. The small parts being die-casted weigh less than one gram each.

Pumps

Milton Roy Company, 1300 E. Mermaid Lane, Philadelphia 18, Pa., is producing a new Model CM chemical metering controlled volume pump. Typical appli-

(Continued on Page 96)



— Mr. L. P. Vollmuth, V. P. of Bart Mfg. Co., Belleville, N. J. talks acid tank protection:

"Bitumastic coatings have given us 2½ times the service life of more expensive materials"

Bart Manufacturing Co., producers of "Lectro-Clad" plate, pipe fittings and other electroplating processes, has a really tough corrosion problem on plating and pickling tanks. Protective coatings must withstand the fumes and occasional spillage of hot sulfuric acid and alkaline cleaning solutions. An expensive, complex coating system applied to the submerged tanks failed in just one year.

The tanks were removed from their pits, cleaned and given two coats of Bitumastic No. 50, the heaviest of the coal-tar based Bitumastic coatings. That was back in 1956 and on recent inspection, the Bitumastic coating system is still sound with no sign of deterioration. Bart makes extensive use of Bitumastic coatings in its other plants, too, where protection of chemical equipment is required.

Our files are filled with case histories like this, where firms have found coal-tar based Bitumastic coatings the answer to tough corrosion problems. We'd like to send you more information for your evaluation of these materials; it is likely they can help in *your* corrosion-prevention program as well. Use the coupon below, or write Koppers Company, Inc., Tar Products Division, Pittsburgh 19, Pa.

District Offices: Chicago, New York, Boston, Los Angeles, Pittsburgh and Woodward (Birmingham) Alabama.

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COATINGS AND ENAMELS

NEW PRODUCTS

(Continued From Page 94)

cations are for metering additives, dyes, corrosion inhibitors, glues, dyes, wet strength resins and corrosive liquids against a head.

•

Sutorbilt Impellers on gas pumps are available with chemically deposited nickel plated coatings for corrosion resistance. The pumps are designed for sewage treatment and chemical plant applications. More information is available from Sutorbilt Corp., 2966 E. Victoria St., Compton, Cal.

Rectifiers

Harco Portable Model No. 3-9 test rectifier provides adjustable direct current output on a timer-interrupted cycle or continuously to determine protective current requirements for cathodic protection. Designed to operate from a 115-volt a-c source or gasoline motor generator, the rectifier has a rated output of 54-volt, 20-amperes for intermittent use or 54-volt, 10-amperes in continuous operation. Details are available from Harco Corp., 4600 East 71st St., Cleveland 25, Ohio.

Surface Preparation

Scotch-Brite, a finishing material made of nylon web impregnated with fine grades of abrasive mineral, cuts pickling time and produces cleaner steel when used before and during the pickling process, according to Minnesota Mining and Manufacturing Co., 900 Bush Ave., St. Paul 6, Minn. The material is supplied in disc form.

Testing

American Research Corp., Farmington, Conn., has published a 12-page booklet on environmental test equipment. Altitude and temperature-humidity walk-in rooms, large sand and dust chambers, explosion testing chambers and rain-sunshine testing equipment are described.

•

HSP-909, a new bulletin describing equipment and reagents for rapid, field water testing is available from Hagan Chemicals & Controls, Inc., P. O. Box 1346, Pittsburgh 30, Pa.

MEN in the NEWS

Woodrow L. Clark has been appointed sales representative of Prufcoat Laboratories Inc., for the Pittsburgh and Charleston and Huntington, W. Va., territory.

•

Oscar H. Hunter, Jr., has been appointed manager of the new Field Services Department of Nowery J. Smith Company.

•

H. M. Jones is the Houston representative for Perrault Equipment Company, Inc.

Ernest C. Ladd has been transferred to the Technical Department, Fibers Division of American Viscose Corp., Fredericksburg, Va., as water and waste control chemist.

•

J. Earl Taylor has been appointed as manager of research and development for Girdler Catalysts, Louisville, Ky., a unit of Chemetron Corporation's Chemical Products Division.

•

William R. Lucas has been appointed regional chemicals sales manager for Atlas Powder Company in Chicago. **Edward M. Simon, Jr.,** will be his assistant.

•

R. T. Thompson, sales engineer for Tretolite Company division of Petrolite Corp., Houston, has retired.

•

Richard R. Vehr has been promoted to sales manager of the Dallas branch of electrical products division of Minnesota Mining and Manufacturing Co. **Robert W. Rayburn** was promoted to sales manager for the San Francisco-Seattle area.

•

Fred H. Meyer, Jr., NACE member, has accepted the position of senior engineer in the processing department at Aeronca Mfg. Co., Middletown, Ohio.

•

George F. Paulus, NACE member, has joined the research and development staff of Acheson Colloids Co., Port Huron, Mich., as a product development chemist.

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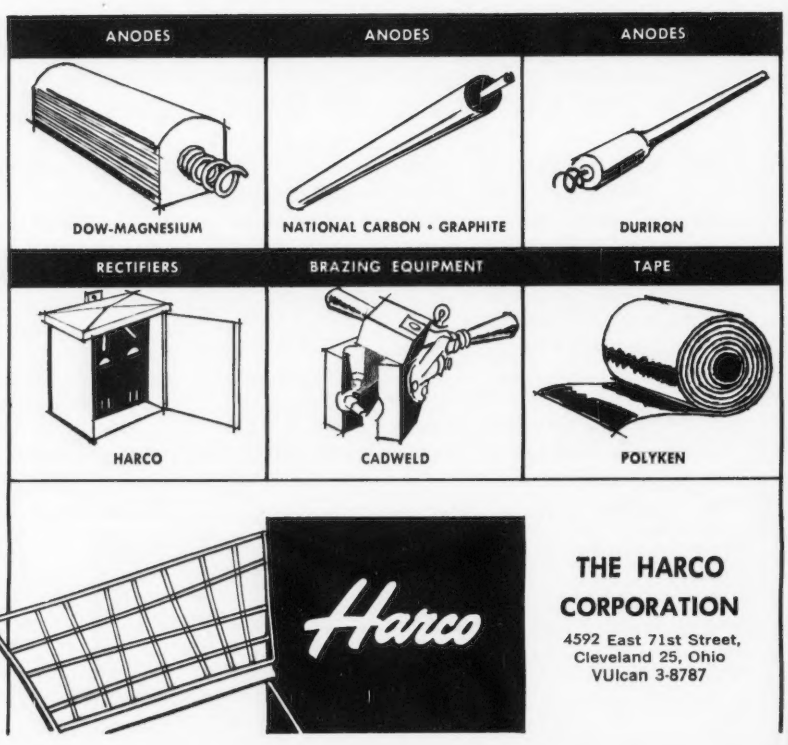

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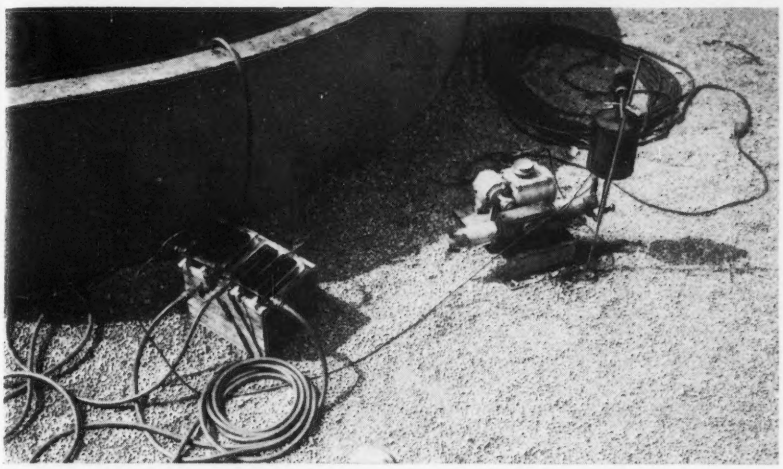


Figure 1—Field installation showing screening test set up for one inhibitor.

Abstract

A method for screening water flood corrosion inhibitors which uses the actual field system is proposed. The method uses two plastic cells, as supplied by National Aluminate Corporation in series with chemical injection made at the exit of the first plastic cell. There were 25 feet of garden hose between the two blocks. Coupons and electrical resistance probes were used to determine the effect of the inhibitor. Good correlation was obtained between the screening test and a field trial of the same inhibitor. 5.8.1

Continuous Injection Required

The key to successful use of the field method of evaluating corrosion inhibitors lies in continuous injection of the proper amount of inhibitor so the stream of water has the desired concentration of inhibitor at all times. Figure 4 shows the detail of the injector. Original tests with this equipment used one half of a pump equipped with 3/16 inch plungers. The normal operating range of this pump required that the inhibitor be diluted with volumes of water in order to be able to inject 20 ppm of chemical into water flowing at five liters per minute. See Table 1.

It was found that some inhibitors being tested would not make a stable dispersion in four volumes of water.

(Continued on Page 98)

A Field Method

Screening Inhibitors for Prevention of Water Flood Corrosion★

Introduction

STARTING POINT for a new system designed to screen water flood corrosion inhibitors was provided by T. R. Newman's[1] development of the recirculation test using plastic cells.

This procedure involved taking a stream of water to be tested off the main source in an operating system and passing it successively through two plastic cells with a chemical injection being made between the two cells. Discharge from the second cell is to waste.

A 25-foot length of garden hose inserted between the injection point and the test cell was to insure sufficient time of contact between the inhibitor and the water so that the inhibitor was dispersed uniformly.

Figure 1 and 2 show typical field installations of the screening test. Figure 3 shows the plan of the test installation.

Each cell was set up to contain one polished coupon, one sandblasted coupon and one electrical resistance probe.[1-6]

The typical two week test period may be shortened or extended as circumstances dictate.

Advantages of System

This unit was designed to overcome valid objections that laboratory results may not be duplicated under field conditions. Conventionally in the laboratory, tumbled closed systems or recirculated closed systems are used.[1] In these systems the water usually is treated once with the desired quantity of inhibitor.

It is believed the field method proposed here produces answers which are more reliable than those derived from laboratory tests. Inhibitors exhibiting poor or slow dispersibility will perform poorly in field test equipment, whereas they may show excellent results in the laboratory where the constant recirculation or agitation insures complete dispersion. The field method has additional advantages in that it utilizes water which is ultimately to be treated under conditions of temperature, pressure (low pressure side of system) and dissolved gas content prevalent in the system.

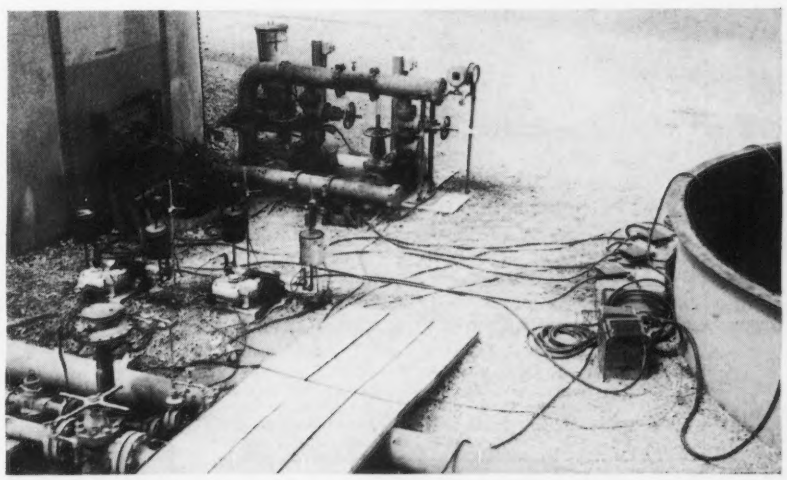


Figure 2—Field installation showing screening test set up for simultaneous screening of four inhibitors.

★ Revision of a paper titled "Corrosion Measuring Techniques for Use in Water Flood Systems" by Charles C. Wright, Oil Well Research, Inc., Long Beach, Cal., presented at a meeting of the Western Region, National Association of Corrosion Engineers, November 17-20, 1958, Los Angeles, Cal.

Screening Inhibitors—

(Continued From Page 97)

These inhibitors were handled satisfactorily by installing a slow speed mixer to keep fluid in the reservoir constantly in motion. Other pumps being considered have an operating range which will permit injecting undiluted inhibitor.

Electrical Resistance Measurements

The electrical resistance probe used is made of mild steel. Probes were installed as received with no further surface preparation. Probes were used as a means of indicating the changes in corrosion rate and not to measure magnitude of attack.

Experience with them shows that a new probe requires 9 to 12 days' exposure before the corrosion rate slope levels off in the oxygen-free Gaspur zone water (see Table 2 and Figure 5). This "activation" period may be caused by traces of hydrocarbon on the probe surface, by slight oxide film, by time to obtain bacterial growth or by other causes not recognized at this time.

The probe was mounted in the plastic cell by means of a ¼-inch copper tubing compressing fitting drilled out to the diameter of the probe. An O-ring gasket was substituted for the brass ferrule.

Manner of Using Coupons

The corrosion coupons used in this study were prepared in the manner suggested by Watkins.[7] They were made of 0.025-inch steel shim stock. One coupon of each set was sandblasted and the other was polished. Coupons were preetched and cleaned as suggested by Watkins before use.

The polished coupon permits examination for evidences of pitting, while the sandblasted coupon provides a more active surface for general corrosion.

Coupons were held in the plastic cell flow channels by inserting them in the slit end of a ¼-inch diameter plastic rod, which in turn was mounted in the block in the same manner as the probes.

Test Run Data Given

A test run was made using oxygen-free Gaspur zone water from the Wilmington oil field (chemically similar to sea water) flowing at the rate of 5 liters per minute.

(Continued on Page 110)

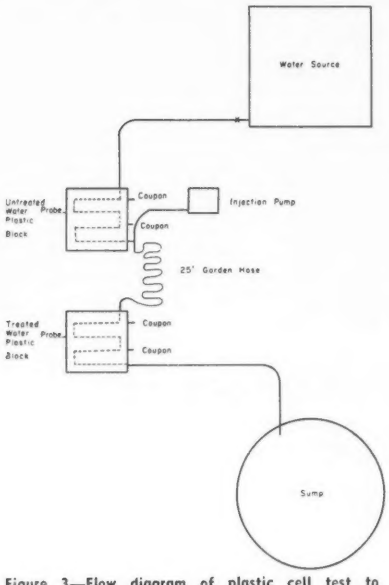


Figure 3—Flow diagram of plastic cell test to evaluate corrosion inhibitors.

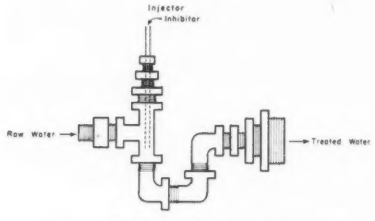


Figure 4—Diagram of a chemical injector.

TABLE 1—Chemical Injection

	Chemical required for 5 liters per minute water flow					
ppm desired	5	10	15	20	25	30
ml. per minute	0.025	0.050	0.075	0.100	0.125	0.150
ml. per hour	1.50	3.00	4.50	6.00	7.50	9.00
ml. per day	36	72	108	144	180	216

TABLE 2—Gaspur Zone Water Analysis

Cation		Anion	
Sodium...	8,978 mg/l	Chloride	16,460 mg/l
Ammonium	10.1	Sulfate	1,921
Calcium...	438.4	Bicarbonate	392.6
Magnesium...	1,189	Borate	11.3
		Sulfide	0.71

pH 7.6
Free Carbon Dioxide 40 mg/l

TABLE 3—Summary of Test

Exposure Time	Chemical Injection	Corrosion Rate, Mils/Year		
		Probe	Polished Coupon	Sandblasted Coupon
14 days...	19.2	0.04	0.2	0.4
14 days...	none	1.4	48.6	57.1

TABLE 4—Comparison of Sensing Units in Plastic Cell With Sensing Units in a Treated Field System

	Corrosion Rate, 15 days exposure	
Corrosometer Probes:		
Plastic Cell.....	2.5 μ inches or 0.06 mpy	
Field System[1].....	0 μ inches or 0 mpy	
Corrosion Coupons:	**Polished**	**Sandblasted**
Plastic Cell.....	0.3 mpy	0.8 mpy
Field System.....	0.7 mpy	0.8 mpy

[1] Reading fluctuations were greater than the total reading change on the probe in the plastic cell

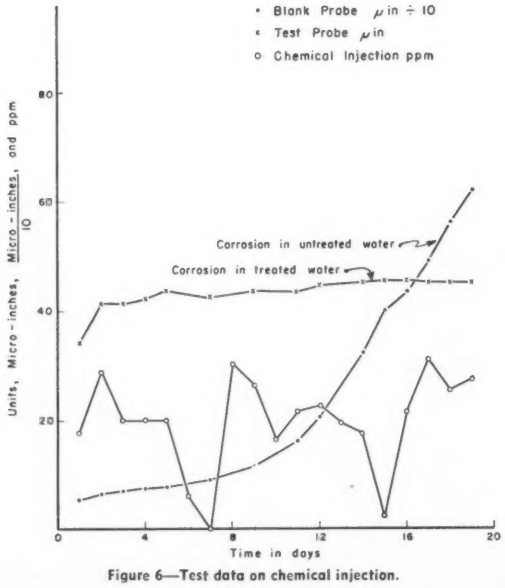

• Blank Probe μin ÷ 10
× Test Probe μin
○ Chemical Injection ppm
Units, Micro-inches, Micro-inches, and ppm
Corrosion in untreated water
Corrosion in treated water
Time in days

Figure 6—Test data on chemical injection.

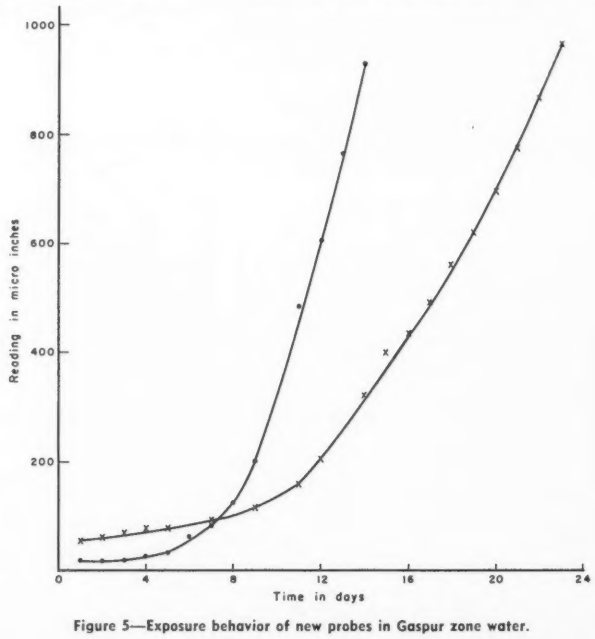
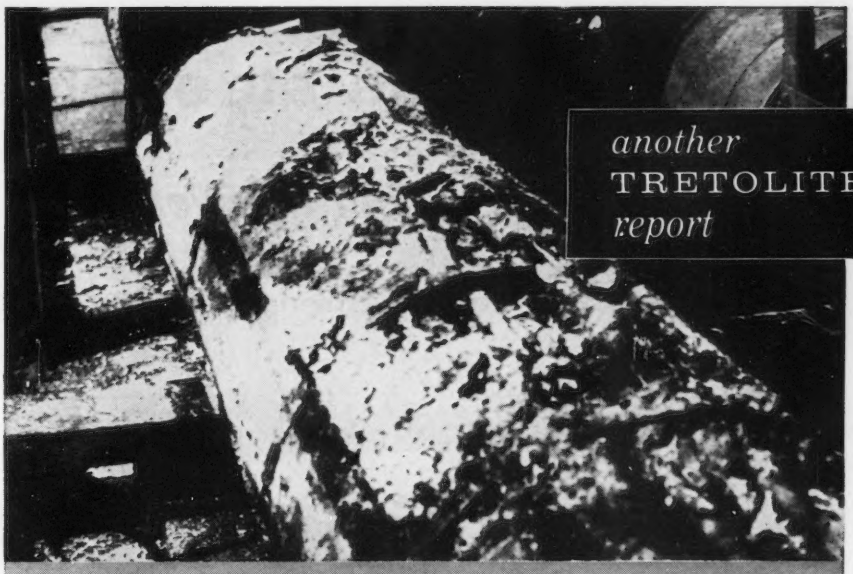


Figure 5—Exposure behavior of new probes in Gaspur zone water.



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TRETOLITE SERVICE
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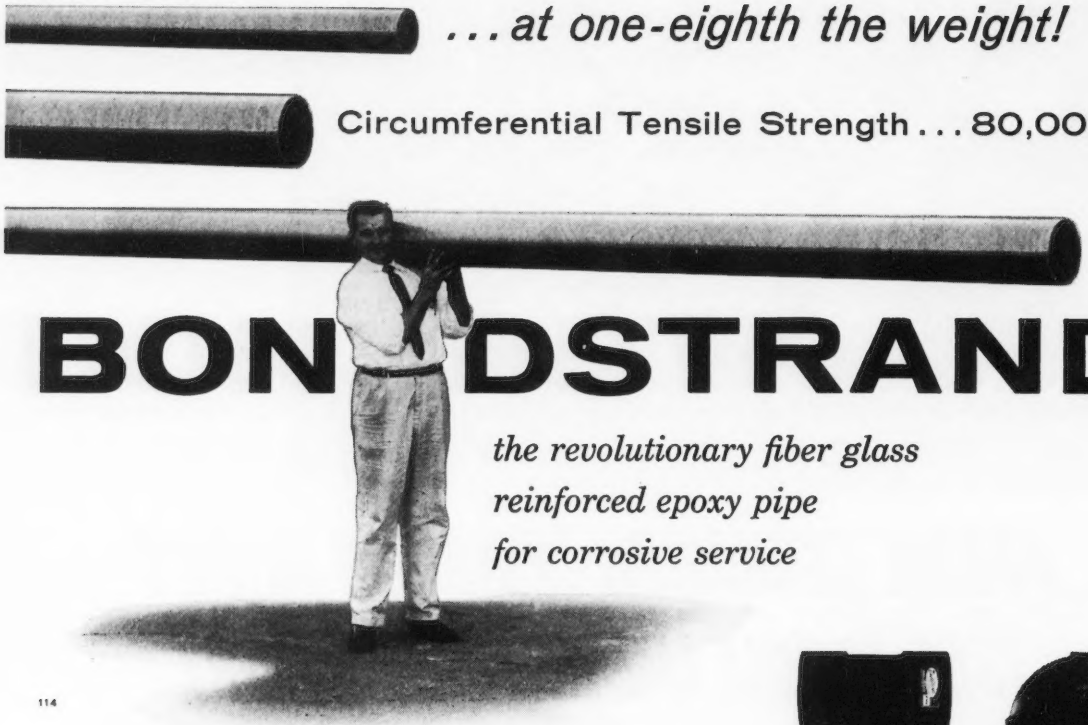

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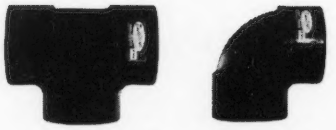

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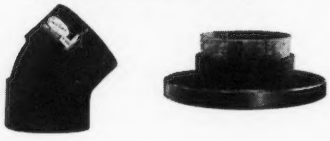

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Use of Plastics and Synthetic Elastomers for Underground Coatings*

Traditional Coatings—Coal Tar and Asphalt

DURING THE nearly 200 years that coal tar has been used successfully as a coating material, a majority of the underground steel pipe lines in the United States has been coated with this material. Several properties make it suitable for coatings, stability, chemical inertness, low moisture absorption and permeability, adhesion to metal, relative ease of application and low cost. The lower cost of asphalt recently has caused its increased use.

In a 350-mile installation by Natural Gas Pipeline Company of America, estimated costs for protective materials (including wrappings) on 26-inch pipe were $1200 per mile for asphalt and $1700 per mile for coal tar.[1]

Specifications[2] have been written recently by NACE to standardize asphalt protective coatings.

Coal Tar and Epoxy Resin Coatings

Although coal tar and asphalt have been used mainly for underground pipe coatings and are standard materials because of their long successful service and good properties, these coatings have some major disadvantages listed by Shideler and Whittier:[3]

1. Large amount of major equipment required for application.
2. Hazards of working with molten materials at temperatures to 500 F.
3. Inability to resist effects of soil stress, mechanical damage and danger of puncture by hard objects in the backfill.
4. Necessity for primer, requiring double handling of pipe and careful coordination between application of primer and enamel to obtain maximum adhesion.

Shideler and Whittier also report that coal tar-epoxy compositions are promising materials which may overcome the disadvantages of coal tar coatings.

Many synthetic resins tried in coal tar coatings are incompatible with coal tar. Others have disadvantages such as production of undesirable by-products during cure.

Epoxy resins, introduced about 10 years ago, can be cured at room temperature, are compatible with coal tar and produce no undesirable by-products on cure. These resins are condensation products of a dihydroxy phenol and epichlorhydrin. Low molecular weight resins are liquids, but higher ones are solids. The resins are polyethers containing polar hydroxyl groups which are thought to be responsible for their excellent adhesion. Low molecular weight epoxy resins are preferable for combination with coal tar because of better compatibility, low viscosity and greater concentration of active epoxide groups.

Agents are available for either room temperature or high temperature cure. High temperature curing can be used if heat is applied to the pipe before coating application.

Over six years of experimental and field testing have resulted in a typical formula: 30 percent epoxy resin, 25 percent coal tar, 20 percent solvent and 25 percent pigment. Coatings of this type have been used as industrial coatings and to a limited extent in underground service since 1953.

After addition of the curing agent or catalyst, the reaction is exothermic. Pot life varies from one-half hour to eight hours at temperatures from 40 to 120 F. The epoxy resin and catalyst are supplied in different packages for mixture just before use. Coatings may be applied by brush or roller. For spray application, either addition of thinner or the use of very high pH should be added to avoid the need of high pressure.

Sufficient setting or hardening of the coating to permit handling of pipe without damage may require from a few minutes to a few hours depending on the temperature of the pipe and the catalyst used. Ultimate chemical and physical properties may not be attained for a week.

(Continued on Page 102)

Abstract

Traditional coatings of coal tar or asphalt and combinations of coal tar with epoxy resin are discussed. Application problems are given briefly. Polyvinyl chloride and polyethylene tapes, their characteristics and service as underground coatings are presented.

New materials that have characteristics which make them usable as underground coatings are given with their physical properties, resistance characteristics and costs. Included are neoprene, chlorosulphonated polyethylene, butyl rubber, polysulfide elastomers, butoxy resins, fluorine containing polymers and silicone rubbers. 5.4.10

★ Extracted from a paper titled "New Materials for Underground Coatings" by E. G. Partridge, University of Southern California, Los Angeles, Cal., presented at a meeting of the Western Region, National Association of Corrosion Engineers, November 17-20, 1958, Los Angeles.

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Underground Coatings—

(Continued From Page 101)

Properties of Cured Coal Tar-Epoxy Films

These coal tar-epoxy films adhere to metal pipe so well that the coating can be removed only by sand or grit blasting. Coated metal may be bent. Adhesion is retained even when the coating is immersed in water or aqueous solutions. The coating does not crack at —40 F and can be bent at fairly low temperatures without damage. Continuous exposures to 300 F temperatures will not damage the coating: it hardens at higher temperatures.

Chemical resistance of the coating is between that of the coal tar and the epoxy resins: fairly resistant to strong mineral acids (not including nitric) and alkaline solutions and not affected by crude petroleum or petroleum products. It is not completely immune to aromatic solvents but resists them better than do other bituminous coatings.

These coatings have electrical resistance over 10 megohms per square foot for 20-mil coating. Service also shows good electrical properties.

Application Problems

Application problems, mainly arising from setting time after application, are being overcome by using preheated pipe and by modifying the coating and catalyst.

A thin film of coal tar-epoxy resin coating was successfully applied on the job to 50 miles of 3 to 16-inch line in a gas gathering system by Trans-Continental Gas Pipe Line Company.[4] Average thickness was 9 to 10 mils applied in one pass with no outer wrap. The two components were pumped from drums through a preheater before mixing. Tests have indicated little damage to the coating in handling, and it was to be handled as bare pipe during construction of the line.

Because coal tar has a record of successful use for a long period and epoxy resins have given good service both above and below the ground for over ten years, the combination probably will have a long service life.

Other possibilities for additives for coal tar or asphalt to increase strength and improve properties are natural or synthetic rubbers which have been used in road construction. One synthetic rubber is butyl rubber which has excellent aging and chemical resistance properties and is relatively low in cost (about 25 cents per pound).

Plastic Tapes

In recent years, plastic tapes of different types in combination with other materials have been used to protect underground lines from corrosion.

One combination was a 1/32-inch coating of microcrystalline wax as a pipe coating and a 2-mil polyvinylidene chloride wrapper as a protective film. Microcrystalline wax has been used over 30 years to protect underground pipe because of its excellent wetting, impermeability and stability properties, but it is not strong enough to withstand soil pressures. The combination with the protective plastic film has given excellent results in installations dating back to 1950. Cost of these materials is comparable with other materials commonly used at present.[5]

Another combination was a layer of petrolatum combined with glass fiber fabric and a backing of polyvinyl chloride film. This tape gives a coating of about 0.036 inch and is being used more, particularly for abrasive conditions.[6]

In the United States, pressure sensitive tapes consisting of polyethylene or polyvinylchloride with an adhesive coating are being considered. These tapes can be hand or machine applied to pipe. This tape coating has overlapping tape layers for good adhesion to coated or bare pipe and also to each other and thus provides a protective layer against corrosion.

Both polyvinyl chloride and polyethylene are suitable for coating service, but there are differences as discussed below.

Vinyl chloride is unsaturated and contains a reactive double bond. However, in formation of the polymer, the double bond is lost. The chain is saturated and therefore chemically inert and resistant to many chemicals including oxidation and aging influences. Under the influence of heat or light, polyvinyl chloride discomposes slowly to evolve hydrogen chloride and also loses some of its properties. Chemical stabilizers are normally added to prevent deterioration. For desired plastic properties in polyvinyl chloride, plasticizers are added to polyvinyl chloride powder. Physical properties, particularly hardness, modulus and chemical resistance depend on the amount and kind of plasticizer used.

A danger in polyvinyl chloride tapes coated with adhesives is that migratory plasticizers may diffuse into the adhesive layer and destroy the bond. Another deteriorating influence is bacterial action which may take place with certain plasticizers. This can be controlled by choice of plasticizer and use of bactericides.

Polyethylene, like polyvinyl chloride, has good physical characteristics of strength, resistance to tear and abrasion and resistance to many chemicals and deteriorating influences. Permeability, particularly to water vapor, is very low. Because of branching in its molecular chain polyethylene oxidizes readily at the branch point under the influence of heat or ultra-violet light and loses mechanical strength.[7] The effect of light can be counteracted by addition of a small amount of carbon black to the polyethylene compound; effect of heat can be counteracted by certain antioxidants.

Polyethylene and polyvinyl chloride tapes have given good results in service tests. While properties of the tapes depend on base polymer and added ingredients, polyethylene tape is generally harder, higher in modulus and lower in permeability to moisture than polyvinyl chloride tape. Cost of base standard polyethylene is about 35 cents per pound; high density type is about 43 cents per pound. Base standard polyvinyl chloride costs about 25 cents per pound.

Because of its usually lower modulus and lower hardness, polyvinyl chloride tape is generally easier to apply to irregular shapes such as tees and other fittings than is polyethylene, but is more likely to harden in use.

Commonly used sealants and adhesives combined with these tapes are based on polyisobutylene or butyl rubber which is polyisobutylene containing about 3 percent isoprene units. Polyisobutylene is a saturated polymer which is resistant to many chemicals and ages very well. Its cost is 35 to 45 cents per pound. Butyl rubber, because of the isoprene units present, is capable of vulcanization but also is more reactive. Its cost is about 25 cents per pound.

Chief advantage of the plastic tapes over coal tar and asphalt coatings is lower application cost. Application properties, particularly at joints and fittings, and resistance to mechanical damage during installation and back-fill are also satisfactory. In the Natural Gas Pipeline Company experiment, plastic tape installation cost was estimated to be about 55 percent of that for coal tar or asphalt coating, making plastic tape competitive on an over-all cost basis although the materials cost is about double that of coal tar (including wrappings).[1] In field coating, particularly of joints, tees and other fittings, proper application of coal tar or asphalt is difficult to achieve. A more uniform coating can be more easily obtained with a tape wrapping. Also, tapes are resistant to abrasion and mechanical abuse.

Because properties of plastic tapes vary greatly with the type of base plastic used and because plasticizers, pigments and anti-oxidants are added, tapes should be purchased under specifications, and control tests should be established. Suggested properties which should be specified and tested are hardness, tensile, elongation, modulus, permanent set, heat and light aging, resistance to moisture, oils and tear and moisture vapor transmission.

Materials Used in Similar Service

Other materials such as neoprene, chlorosulphonated polyethylene, butyl rubber, polysulfide elastomers and butoxy resins have not been used extensively as underground pipe coatings but have excellent properties and have been used in similar services.

An elastomeric polymer of 2-chlorobutadiene (chloroprene) used in the rubber industry, has these physical properties: resistance to many chemicals including oils, low permeability to moisture, good aging properties including resistance to oxidation, sunlight and ozone, resistance to bacterial action and flame resistance.

Because of these properties, this polymer has been used commercially for over 15 years as a coating to protect lead sheathed cables from corrosion and mechanical damage. Most of the cables protected are installed in ducts under city streets where they come into contact with a variety of elements. The coating must withstand severe mechanical abuse during installation. As a result of satisfactory service, tentative specifications have been issued recently for neoprene protective coverings by the Association of Edison Illuminating Companies.

Coatings made of this polymer have been used successfully to protect bare cables in sea water.

An example of property retention by a neoprene compound buried in soil for seven years is given in Table 1.

(Continued on Page 104)

TABLE 1—Neoprene Compound Property Retention.*

	Modulus at 300%	Tensile	Elongation
Original.....	450 psi	1200 psi	755%
After 7 years..	970 psi	1340 psi	490%

* Compound was buried in soil for seven years.

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Underground Coatings—

(Continued From Page 102)

Like other base materials, neoprene must be compounded and cured properly. Protective materials must be included for protection against aging and to give chemical stability; without these, slow decomposition takes place and hydrogen chloride is evolved with accompanying deterioration in physical properties.

Commonly used base neoprene costs 41 cents per pound, but the price of the finished coating depends on other ingredients added and processing costs.

Chlorosulphonated Polyethylene

A material recently developed is chlorosulphonated polyethylene, a protective coating resistant to many influences which deteriorate most polymeric materials. Vulcanizates of this material are inert to ozone attack and are resistant to weather and many chemicals. They are serviceable at high temperatures and are resistant to flame and bacterial attack.[8,9]

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Because of its excellent electrical properties, resistance to moisture, and retention of properties under adverse conditions, chlorosulphonated polyethylene covered wires have given excellent results in experimental installations. In one instance, coated wires buried in top soil over a year showed no change in tensile and elongation and showed good retention of electrical properties. At present, this material is relatively expensive (about 72 cents per pound).

Butyl Rubber

Because of its combination of properties and its resistance to soil acids and bacterial action, butyl rubber should also be considered for other coatings uses beyond its present use as sealant and adhesive on plastic tapes. It has been used as an impregnant for burlap and as coating for the ground in waterproofing irrigation ditches. After 13 years of service, the butyl rubber has retained 90 percent of its original physical properties. It has good aging and chemical resistance characteristics and costs only 25 cents per pound.

An interesting use of a butyl rubber base in solvent has been as an emergency sealer of leaking gas pipe by filling the pipe with the paint emptying and then passing air through to evaporate the solvent.

Polysulfide Elastomers

Polysulfide elastomers are resistant to oils, solvents, weather and ozone and impermeable to gases, water vapor and solvents. Although they have relatively poor physical properties, they are adequate for many uses. The properties are retained for long periods in oil immersions and during exposure to weather and to low temperatures. Disadvantages are disagreeable odor, poor heat resistance, poor abrasion resistance and high cold flow. Most commercial polysulfide elastomers cost from 50 cents to $1.35 per pound.

Butoxy Resins

A coating based on butoxy resins derived from styrene butadient copolymers of low molecular weight and high unsaturation has been developed. It has good physical characteristics and chemical resistance, can be applied by spraying and flame cured in a few seconds to give a tough film. A long range estimated price for such resin coatings is 24 cents per pound for a 50 percent solution.[10]

Other Materials

Considerable research is being done in modifying existing polymers and in developing new ones resistant to chemicals over a temperature range from —160 F to 1000 F. These materials, including fluorine containing polymers, silicone rubbers and modifications, are being considered for use on aircraft and missiles.

Although these materials are too expensive for consideration as pipe coatings, they may be practical in cases in which unusual properties are required.

Fluorine Containing Polymers

Fluorine containing synthetics have outstanding resistance to chemicals and high temperatures plus good physical properties. One of these polymers is a polytetrafluoroethylene which has been used at temperatures from —450 F to 500 F. It is extremely resistant to chemicals. The only chemicals known to affect it are molten alkali metals, fluorine at elevated temperatures and pressures and certain complex halogen compounds such as chlorine tri-fluoride. It also is resistant to moisture, unaffected by outdoor weathering and has excellent electrical properties. Its cost is about $4.50 per pound. Since it is not possible to make many kinds of articles from this material, similar materials with better processing properties have been developed.

A copolymer of monochlorotrifluoroethylene and vinylidene fluoride can be vulcanized, is easier to process than polytetrafluoroethylene and retains much of its chemical resistance, but it is more expensive ($10 to $15 per pound).

Another member of the polymer series is a linear copolymer of vinylidene fluoride and hexafluoropropylene that retains much of the chemical resistance of the other polymers but is easier to process. It can be handled on conventional rubber processing equipment. It also is resistant to chemicals such as fuels and hydraulic fluids. Its cost is about $15 per pound.

Silicone Rubbers

Silicone rubbers are another material which can be considered for coating purposes. These rubbers have good aging characteristics, are resistant to many chemicals and oils and can be used at temperatures from —160 F to 600 F.

These rubbers are relatively low in physical properties although progress is being made to improve compounding and vulcanizing. Cost is from $2 to $5 per pound.

Modifications of silicone rubbers are being tested to improve their physical properties and particularly their resistance to chemicals such as rocket fuels and hydraulic fluids while retaining a wide temperature range in which they can be used.

One line of research is the replacement of organic groups in silicone rubbers by others. For example, fluorosilicone rubbers have been developed in which some of the organic groups are replaced with fluorine to obtain some of the chemical resistance exhibited by fluorine containing elastomers while retaining the temperature range of usefulness of silicone rubbers.

An even newer approach to the problem is the substitution of some cyano or nitrile groups for organic groups in silicone rubbers to attain oil and solvent resistance of nitrile type synthetic rubbers without losing temperature range.

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FIGURE 1—Stress corrosion cracking apparatus used at the National Bureau of Standards. A magnesium alloy specimen inside a cell (left) is stressed. A corrosive medium can be added before or after the stress is applied. Distance through which the specimen extends is indicated by a dial gage (right). From a correlation between wire strain gage readings on the specimen surface and the dial gage readings, strain rate is calculated from dial gage readings taken at stated intervals. Strain rate was shown to be an important factor in stress corrosion cracking.

Mechanism of *Stress Corrosion Cracking*★

Abstract

The mechanism of stress corrosion cracking in AZ31B magnesium alloy was investigated in an aqueous solution of 3.5 percent sodium chloride plus 2.0 percent potassium chromate. Specimens immersed in the corrodent and extending at an average rate of 500 ± 100 microinches/inch/min., one minute after the stress was applied generally failed within 5 minutes. For slower rates, specimens did not fail in 1000 minutes. Stress corrosion cracking could be stopped by application of a cathodic current of 0.5 ma/cm² of exposed area. Experiments indicated that stress corrosion cracking of AZ31B magnesium alloy is primarily an electrochemical process and is dependent on strain rate. 3.2.2

CLEARER UNDERSTANDING of the mechanism of stress corrosion cracking is the objective of a current research program sponsored jointly by the National Bureau of Standards and the Corrosion Research Council. As part of this work, Hugh L. Logan of the Bureau's corrosion laboratory recently completed a stress-corrosion study[1] of AZ31B magnesium alloy (aluminum 3 percent, zinc 1 percent.)

Stress corrosion cracking of metals is recognized as a serious economic problem. Stress corrosion cracking of brass (season cracking) long has been known to be a potential cause of failure in brass articles formed by spinning or deep drawing and not subsequently annealed. Many failures of riveted steel boilers have been attributed to stress corrosion, known in this instance as caustic embrittlement. Fabrication of steel vessels by welding has reduced, but by no means eliminated, stress corrosion cracking in low carbon and low alloy steels.

Metallurgists and management in the chemical and petrochemical industries have learned in the past few years that many if not most of the leaks that develop in stainless steel equipment used in those industries are in reality stress corrosion cracks. Until such time as the mechanism of stress corrosion cracking is completely understood, methods of mitigation are largely empirical.

Most investigators in the field are agreed that stress corrosion cracking is in part, at least, an electrochemical process. There is wide disagreement, however, as to just how much of the mechanism is electrochemical.

Certain magnesium alloys are ideal for a mechanistic study. Earlier work[2] showed that in the corrodent used in the laboratory (3.5 percent NaCl + 2.0 percent K_2CrO_4) there was a threshold stress for each magnesium alloy. Specimens exposed above this stress failed in a few minutes; below this stress they did not fail in many hours. Hence, many data were readily obtained.

In most of the present work the specimen was placed in a cell (Figures 1 and 2) and the corrodent was added before the stress was applied. While the major strain occurred with the application of the stress to the specimen, the specimen continued to extend, but at a diminishing rate, for a considerable time after the stress was applied. The calculation of many strain rates determined for specimens stressed above, at and below the threshold stress showed that there was a critical strain rate, 500 ± 100 × 10^{-6} in./in./min. as determined one minute after the stress was applied. If this rate was exceeded, early failure by stress corrosion cracking occurred. If the strain rate was less than this value, failure did not occur and the specimen showed little if any general corrosion. Earlier work[3] showed that plastic extension of this alloy ruptured the protective film on it, exposing a film-free surface that was anodic to the normal surface and hence would be attacked by an electrochemical process in an electrolyte. It was also shown that the film tended to re-form. From the present data it was concluded that, if the specimen was extending at an average strain rate in excess of 500 × 10^{-6} in./in./min, there would be certain areas on which the strain rate would exceed the rate of film repair. These areas would be anodic to the filmed surface and stress corrosion cracks would develop. At lower strain rates a protective film would form on the metal and there would be no corrosive attack.

It was assumed that if the specimen was stressed and the strain rate had decreased below the critical value before the corrodent was added the specimen would not fail. Experimentally it was found that in this case the critical strain rate was much lower than that given above. This apparent contradiction in the data was explained when it was noted that there was a marked increase in the strain rate on the addition of the corrodent. This fact also suggested an explanation for a phenomenon previously noted in weather exposure work, namely, most cracking (Figure 3) and failures of specimens were found following rain. If the corrosion products dissolved in rain water had the same effect as the corrodent in the laboratory, increased strain would exist over microscopic lengths of the specimen. Then, if the critical strain rate was exceeded, stress corrosion cracking would occur.

Deviations from the normal strain rate-time curves were found to indicate the formation of stress corrosion cracks. Hence, it was possible to apply cathodic protection to specimens after stress corrosion cracks had developed. Cathodic protection stopped stress corrosion cracking, even after cracks had penetrated an appreciable distance into the metal, by polarizing the entire specimen to the potential of the anodic areas. With removal of cathodic protection the specimen was depolarized immediately and failure by stress corrosion cracking occurred within a few minutes.

These data indicate that the inception and propagation of stress corrosion cracking, in this alloy, is primarily an electrochemical process.

★ Condensed from an article titled "Mechanism of Stress-Corrosion Cracking in AZ31B Magnesium Alloy" by Hugh L. Logan, published in *Journal Research NBS*, **60**, 503 (1958) December.

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Coatings Engineers Help In Power Plant Planning*

Abstract

Advantages gained by the protective coatings engineer in cooperating with architects in power plant design and construction are discussed. By presenting the coatings problems in relation to climate and environmental causes of corrosion, the engineer helps the architect to eliminate coating problems. The inclusion of protective coating specifications and material specifications are recommended on the architect's drawings with detailed specifications added.

During plant construction, the coatings engineer should inspect items delivered for compliance to coatings specifications and for protection against damage during construction.

Coatings maintenance programs are recommended and should be set up in cooperation with operating personnel so that effective coatings can be achieved economically. 5.11

MORE EFFECTIVE protective coatings systems for power plants can be achieved if the coatings engineer works with the architects in the initial planning of the plant, with the contractor during construction and with operating personnel during plant operations.

After the plant site has been determined, the protective coatings engineer can be of considerable assistance to the architects in planning the plant facilities. Because factors such as winds, temperature ranges, humidity, rainfall, air pollution, chemical fume concentrations from nearby plants, fog occurrences and directions from which storms originate are important considerations in protecting a plant from corrosion, the coatings engineer should bring these factors to the attention of the architects.

The engineer can also emphasize the maintenance economies of the enclosed plant in areas of severe exposure or extreme pollution over the open type plant. Initial painting costs of the open and enclosed types are about the same, but coatings maintenance costs for the open type usually exceed those for the enclosed plant. Additional protection is obtained from the entrapped heat, decreasing the relative humidity of the air inside the buildings and decreasing or eliminating possible corrosive effects from moisture in the structure.

Another factor which is frequently overlooked in planning is location of stairways, open sections of the plant and equipment in the areas least exposed to the weather. In any location, certain sides of the plant are subject to greater atmospheric corrosive effects because winds and storms occur more frequently from certain directions than from others. With a little experience, the protective coating engineer can interpret this data to advantage.

Air pollution from nearby plants also must be considered in planning. An enclosed plant in areas of severe chemical air pollution eliminates the need for a more resistant protective coating system. Pollution from nearby plants can contaminate a surface between coats during application of a paint system. Even after careful cleaning prior to application of intermediate and finish coats, contamination may cause poor adhesion and premature peeling or flaking of the paint films.

Structural Design

Helping architects to incorporate structural designs with corrosion control in mind is another assistance the coatings engineer can give to plant planning. He should encourage the use of smooth shapes and suggest that moisture traps, sharp edges and rough weld joints be eliminated to provide surfaces which can be more effectively coated.

Information on Drawings

Entries and references to protective coatings can be indicated to advantage on the plant drawings. The coatings engineer can help the architect by stating a specific composition reference or trade designation when a special material is to be used in conjunction with the coatings system. The desired thickness should also be given. This information helps the fabricator, the inspector and the operating personnel who later may have to replace the material. In case material fails in operation, specific information on plant drawings can help pinpoint the cause and eliminate repeated material and coating failures.

When special materials should be used in plant structures for more effective corrosion control or for more effective surfaces for protective coatings, the engineer must be able to justify the use of the material on a dollar-and-cents basis.

The architect cannot be aware of all problems concerning plants, so often just the presentation of a coatings problem caused by the use of certain materials will convince him that a substitution is needed.

Surface preparation, shop priming and even finish painting should also be specified on the plant drawings. This may be the only opportunity the coatings engineer has to present this subject. Many items are ordered from the drawings only with no specifications; therefore surface preparation must be given. The protective coating system should be indicated, especially the minimum total dry film thickness. Critical areas can be singled out on the drawing to receive special treatment such as back-to-back angles which are difficult to paint properly after assembly.

These specifications on the drawings will help the fabricator, working with his and the purchaser's inspectors, to deliver the item exactly as designed.

Detailed Specifications

Specifications with accompanying drawings for the power plant and equipment offer the protective coatings engineer his greatest opportunities for future maintenance savings. Although drawings may contain abbreviated instructions as to surface preparation, shop priming and protective coating systems (metallic or non-metallic), more details can be given in the specifications. First, the protective coating engineer may define to the fabricator and inspector the exact protective measures necessary. Second, by reference to certain standards such as those available from the Structural Steel Painting Council, proper surface preparation can be emphasized and defined briefly in the specification. Thus, the item can be properly readied for any later application of the protective coating system.

The protective coating system required, whether complete or only shop primed, can also be defined in the specifications. The materials approved for use must be clearly listed. It should be stated whether other products can be approved for use; if not, it is always a good policy to specify that no substitutions are to be allowed. This policy assures the engineer the system he wants and eliminates later controversies as to other approved equivalents.

The method of application may also be clearly stated in the specifications. It is possible to ask for any means deemed necessary and to ask for a few refinements such as extra attention to edges, welds, rivets and areas difficult to paint after assembling or installing. The maximum time between coats must be stated also because care should be exercised between coats in multi-coat systems. Frequently poor adhesion results from carelessness in application or from contamination between coats.

A good policy is to stencil the product names and date of application on the item. This is one more way of being sure the desired protective coating system is applied, and it may also serve as a check on the fabricator. In the case of complete systems being applied in the shop, the field men can use the information in their maintenance plans.

This data can also be of great value to the protective coating engineer at a later date. He can judge the success or failure of his paint system and correct the weak points.

Plant Construction

During the construction of the power plant, the protective coating engineer should assist in inspecting items ordered earlier. Although this is a responsibility of the constructor, the engineer's assistance will be welcomed because everyone is busy during construction. As the plant is erected, the protective coating engineer may serve in this advisory capacity, seeing that the coatings on the building materials and equipment are in good condition when delivered and continue to stay in good condition during storage and after installation.

Many structural items are received shop primed. They should be checked for compliance to specifications. The protective coating engineer may also judge how his specification can be improved, assuring an even better protective coating system in the future.

Any damage to the specified coatings during installation must be repaired,

(Continued on Page 108)

* Extracted from a paper titled "Planned Painting Protects Power Plants" by W. J. Prather, Pacific Gas and Electric Company, San Francisco, presented at a meeting of the Western Region, National Association of Corrosion Engineers, Los Angeles, November 17-20, 1958.

Coatings Engineers—

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and the specification must state whose responsibility these repairs are. Unpainted or abraded areas must be cleaned and primed properly, a problem arising particularly in structural steel erection. Rivets and bolts must be cleaned and primed by the erector. Thus the structural steel is completely shop primed, protected and ready for subsequent painting.

To assure complete compliance with coating specifications, the engineer should inspect all finish-coated equipment upon arrival at the plant site. Many times the equipment may be an assembly line coating of substandard quality. If so, the protective coatings engineer should always bring this to the attention of the construction group for possible adjustments from the supplier. The inspector should also be told of the deficiencies to avoid future repetition of the incident. This sometimes calls for a great deal of tact, but in dealings with the inspection section, no difficulties are encountered whenever the purpose behind the specification is explained and when it is shown that the primary interest is in obtaining the equipment in the best possible condition.

Finally, the protective coating engineer should review his specification, the bills of material or drawings to ascertain that everything was clearly stated. Constructive changes should be welcomed and put into effect.

Many of the deficiencies in protective coatings noted at the construction site may be minimized by helping inspectors during fabrication. This may best be done by prior discussion of the specification and drawings before fabrication begins, moral support by accompanying him to the fabricator or vendor for a preliminary discussion of the protective coating system and by later accompanying him to the fabricator to further check the protective coatings on the finished products. This cooperation among the coatings engineer, the inspectors, and the vendors will result in more satisfactory jobs. The engineer becomes better aware of the problems of both the inspector and the fabricator, and they, in turn, understand the intent of the specifications and drawings. And the protective coating engineer may improve his specifications by learning the weak and strong points in his protective measures.

Frequently the protective coating engineer, as a liaison between operations and engineering design personnel, can accomplish a great deal in continuous follow-up of the operational coating problems. This may seem relatively unimportant, but if neglected, a poor coating system may be used repeatedly later.

Maintenance During Construction

Of great importance also is the maintenance of protective coatings during the construction period. Weak areas in coatings should be remedied as soon as detected. Equipment should be protected by proper care during the time of storage prior to installation and during the time prior to the plant going into operation. This means storage facilities should protect the equipment from the detrimental effects of weather. If not, adequate means such as covering with tarpaulins or plastic coverings will be beneficial.

After installation, considerable damage to the protective coatings on equipment frequently occurs. The coatings must be kept in first-class condition at all times by continuous inspections. If the contractors are responsible, they must bear the cost of repairing unnecessary damage. Thus, the overall final painting costs may be reduced.

Occasionally it is more economical to finish-paint equipment at the fabricator's; in other cases, it may be better to omit the finish coats or any protective coating until just before the plant goes into operation. Each case usually depends upon the type of equipment or the construction conditions. If the protective system can be kept in good condition during the construction period, usually it is good practice to have the coating applied at the vendor's where application conditions are more easily controlled.

Structural steel painting varies also. In most plants, the items are usually delivered blast cleaned and primed. After erection, a spot prime coat, a prime coat and intermediate coat are applied. Finally, as the plant is put into operation, the final finish is applied. This gives maximum protection during the construction period and also a freshly-painted plant at completion of construction. Of great importance is the advantage of reducing the first year's touch-up maintenance. Any weak areas in the protective coating system will have shown up prior to application of the final coat and thus may be corrected.

However, in the case of penstocks, a different procedure has been followed. The sections are lined with coal tar enamel at the fabricator's. On the ex-

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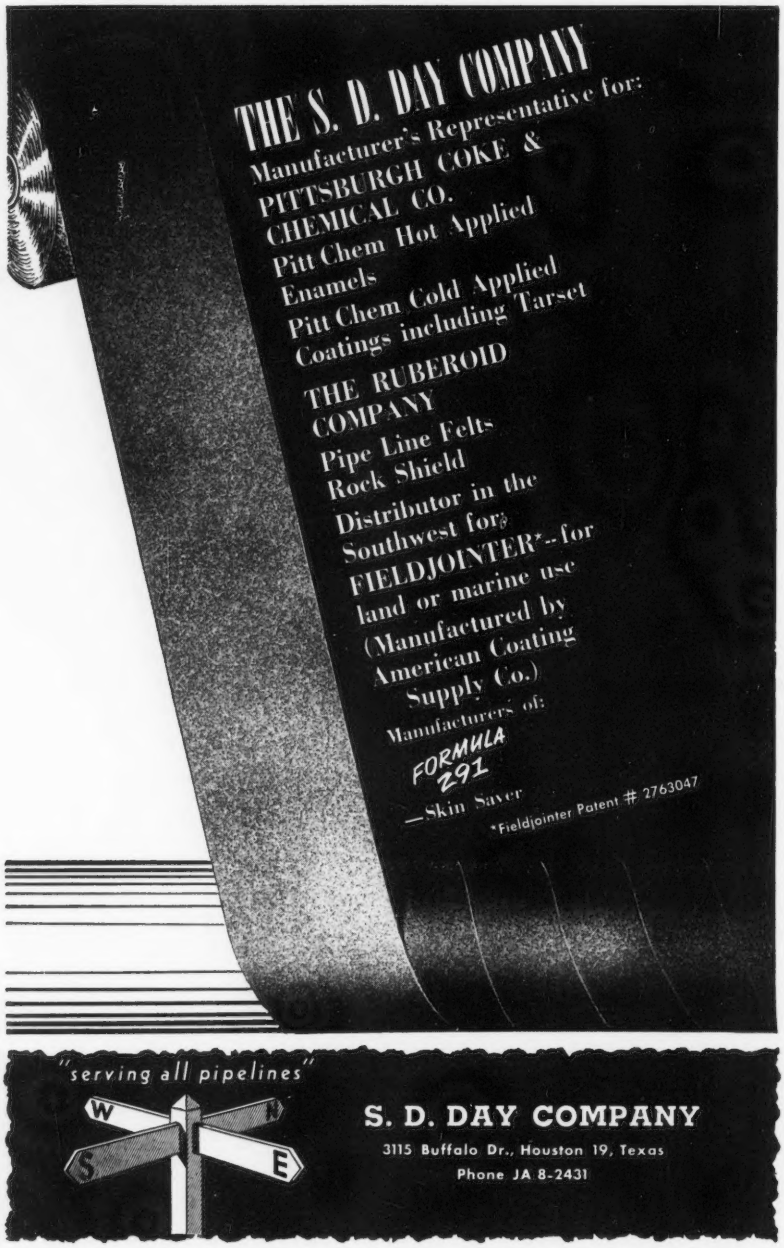

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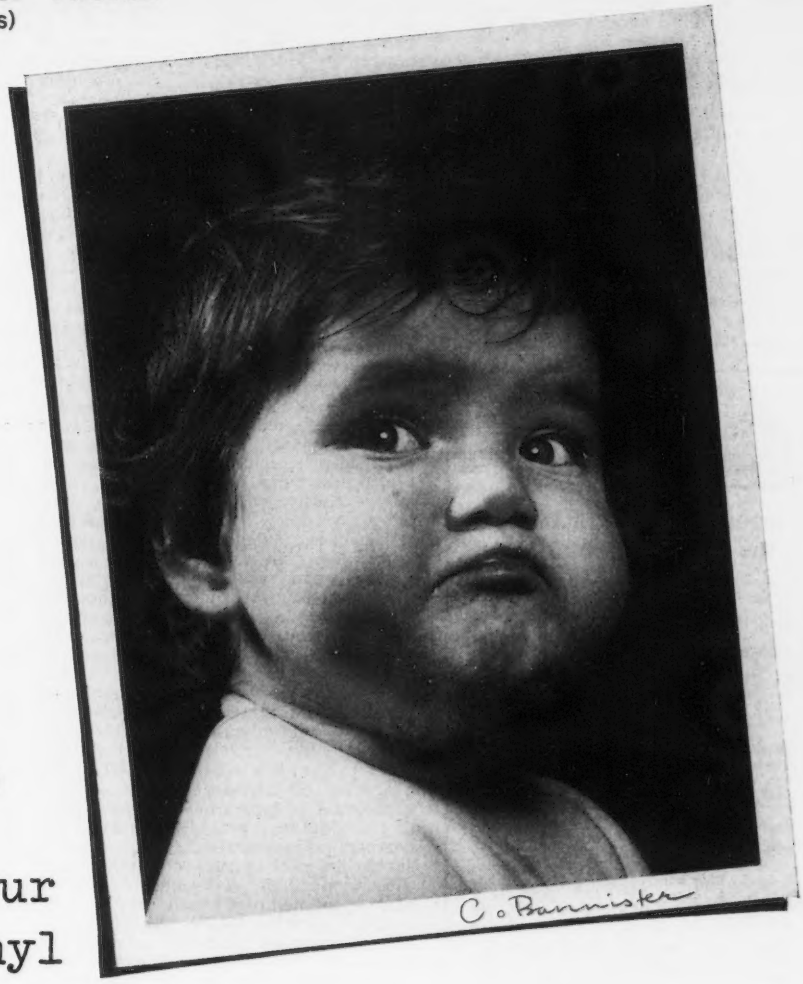

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Coatings Engineers—

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terior, the surface is ordinarily blast cleaned and shop primed. Recently, an additional coat of aluminum paint was also applied at the fabricator's. This system was successful with little touch-up necessary in the field prior to application of a finish coat of aluminum. It may be possible to have the sections completely painted by the fabricator. The only field painting would then be a touch-up of abraded areas and the complete painting of the penstock joints and appurtenances.

While the finish coat is being applied to the structural steel, the finish-up painting is continued throughout the plant. Prior to this time, many items are only shop primed; some, such as piping, brackets and hangers, are still unpainted. Much of the equipment is already finish painted. This finish-up painting completes the protective coating system before the plant is turned over to operating personnel.

Maintenance Program

Once the plant is finished, painted and in operation, a maintenance program should be established by the coatings engineer. He should have a record of all the paint systems used. This record should be compiled carefully for him by the constructor. The engineer should review the systems periodically to obtain information for use in future plant construction.

In order to take advantage of the ground work laid in the original protective coatings, maintenance must be regular and must be performed by well qualified personnel. The original surface preparation must be protected to avoid later expensive surface preparation during maintenance. Frequent touch-up and regularly scheduled overall maintenance accomplish this. In severe coastal exposures, experience in one area on structural steel painting shows the estimated first year maintenance costs to be 6 percent. However, if delayed only one year, costs jumped to 18 percent and to 45 percent the following year. This probably was slightly higher than normal because originally no blast cleaning was accomplished and later maintenance allowed only power cleaning of the steel.

As part of his coatings maintenance program, the engineer should establish a program of laboratory and field tests at the plant to provide information on coatings performance. The test program should define the problem, materials and their application and should include a regular follow-up to reach definite conclusions of value to the engineer. Usually this means that the engineering staff must work closely with paint crews and plant operating personnel.

Maintenance is a continuous coatings problem. In these times of rising costs, the labor costs greatly outweigh the material costs; thus good materials, though costly, are easily justifiable. The coatings engineer must continue looking for more labor-saving means of application such as by hot or cold spraying, roller, or by the use of multi-mil coatings.

Summary

Foresight in the planning, building, and operating of a power plant can pay large dividends in painting maintenance savings. By the protective coating engineer pointing out the influences of geography and climate, the planning architects can avoid many severe atmospheric corrosion problems and the harmful effects of air pollution from nearby industrial areas. Being aware of these facts and watching the actual structural details of the plant itself can minimize many future maintenance headaches.

By careful review of specifications and drawings of equipment during their preparation and by close inspection of equipment delivered to the plant site. The coatings engineer can help in making certain that equipment is properly protected. Quality of materials and equipment may be further assured by close liaison with both the company inspectors and the vendors. Vigilance during the construction period helps maintain the quality of protective coatings.

If constant attention is given to regular maintenance and maintenance methods, protective coatings throughout the plant will remain in good condition at a minimum of expense.

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Screening Inhibitors—

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Chemical injection was planned at 20 ppm and (even though difficulties were encountered with the particular injection pump then in use) average feed was 19.2 ppm. In spite of the irregular feed, readings of the treated water probe showed very little deviation, demonstrating satisfactory inhibitor film retention.

Results are summarized in Figure 6 and Table 3.

Evaluation of the effect of using the plastic cell was made by placing a plastic cell on a by-pass stream (to discharge) at an injection well head. The cell contained the same kind of sensing units as were installed in the field system.

Table 4 presents data obtained during a 15-day period by this method. There is good correlation between the plastic cell and the field system results.

A final comparison was made by taking the inhibitor and injecting it into the field system. Table 5 presents the comparative data showing the correlation between the field test and the screening test.

Correlation between the screening and field tests is good considering that maximum weight losses of the order of 14 mg in two weeks on the coupons are being considered.

TABLE 5—Comparison of Screening Test With Field Performance*

System	Corrosion Rate, Mils Per Year		
	Screening Test	Field Test	
		Plastic Cell	Line
Probe	0.04	0.2	0.5
Polished Coupon	0.2	0.07	0.3
Sandblasted Coupon	0.4	0.2	0.6

* 14-day tests at 20 ppm of inhibitor.

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6. G. H. Rohrback. Resistance Method for Corrosion Measurement. NACE Western Region Conference, Jan., 1958.
7. J. Wade Watkins. U. S. Bureau of Mines R. I. No. 5031.

CORROSION ABSTRACTS

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1. GENERAL

1.2 Importance

1.2.2, 2.1.1

Economic Aspects of Corrosion. A. Keynes. Chemistry and Industry, No. 14, 398-400 (1958) April 5.

Discusses techniques used in economic analysis of corrosion in industry. It is suggested that histogram be constructed showing "corrosion-proneness" of main industries; index of corrosion-proneness is derived simply from relation between Fixed and Total Assets in an industry. For useful comparisons, another histogram is constructed which represents effort or cost which same industries put into fight against corrosion. Factors involved in cost schedule are discussed. Another technique considered is use of indifference curve or iso-quantial analysis. Its use in determining optimum economic balance in protective system using both coating and cathodic protection is illustrated.—INCO. 15931

1.2.5

The Mystery of Pyrophoricity: Metals that Burst into Flame. Richard B. Smith. Am. Machinist, **101**, No. 4, 142-144 (1957).

Problems in handling pyrophoric metals-plutonium, uranium, thorium, zirconium, hafnium, titanium, magnesium, calcium, sodium, potassium-in bulk are considered. Spontaneous fires occur over a wide range of temperatures and storage conditions, including vacuum, argon, and immersion in water. There is evidence that metals are more pyrophoric when they are under stress, when they are contaminated with other pyrophoric metals which lower their melting point and when they contain hydrogen, either from reaction with moisture or because they were originally made from a hydride.—MA 16284

1.4 Bibliographies and Indexes

1.4

Index of Technical Articles. April, 1957, 102 pp. Iota Services, Ltd., 38 Farrington Street, London, E.C.4.

This issue includes a classified list of technical articles published during March, 1957, in about 500 technical periodicals in Great Britain, including 10 daily newspapers. The references consist of the title, a brief abstract and the necessary locator information.

A well-organized index is published both in numerical sequence and in alphabetical order. Abstracts are arranged in numerical sequence according to the index and are easily located. There is an alphabetical author index. 15317

1.4, 6.3.20, 6.3.21

Unclassified U. S. Atomic Energy Commission Reports on the Metallurgy of Zirconium and Hafnium. Richard J. Smith, compiler. Technical Information Service Extension, AEC. U. S. Atomic Commission Pubn., TID-3508, June, 1957, 38 pp. Available from Office of Technical Services, Washington, D. C.

Annotated references to 134 reports are included covering the following aspects of Zr, Hf, and their alloys; physical and chemical properties, phase diagrams, fabrication, testing equipment and methods, and corrosion. (auth.)—NSA. 15434

1.4, 5.9.4

Phosphating Treatments — Patent Literature Survey. E. C. Tinsley. Rock Island Arsenal Laboratory, U. S. Army Ordnance Corps. April, 1957, 148 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington, D. C. (Order PB 131356).

This volume was compiled as an aid to chemists and engineers concerned with production or selection of effective phosphating coatings for metals. Data are given on 522 patents relating to the phosphating process. Each entry is abstracted. A subject index, an author index and a bibliography are also included.—OTS. 16404

1.4, 8.9.5, 8.4.5

Bibliography for Nuclear and Conventional Merchant Ships. F. L. May. American-Standard for the U. S. Atomic Energy Commission. June, 1958, 105 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order ASAE-S-10).

References used by numerous organizations in studies of various phases of the application of nuclear power to merchant ship propulsion are listed in this bibliography. The references are arranged in subject groups which include conventional and nuclear ship characteristics; reactor types; propulsion machinery and auxiliary equipment; economics; basic information on reactor design; radiation and waste disposal; maritime law; and miscellaneous information. Indexes relating each reference to its author and publication number are included.—OTS. 16880

1.6 Books

1.6

Materials Manual. W. D. Valovage and E. E. Baldwin, Editors. Knolls Atomic Power Lab. U. S. Atomic Energy Commission Pubn., KAPL-A-ME-1, Jan. 1, 1954 (Changed from Official Use Only June 11, 1958), 226 pp. Available from Office of Technical Services, Washington, D. C.

The properties of 15 metals and alloys are summarized with bibliographic references. General characteristics such as composition, availability, nuclear properties, and applications are given along with data on fabrication, corrosion resistance, physical properties, and mechanical properties. The metals and alloys treated are: stainless steel types 347, 347(M1), and 304, molybdenum, Inconel-X, beryllium, Carboloy 779,

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Kennametal 138A, sodium, hafnium, zirconium, Zircaloy-II, Cupro Nickel, 2¼ chromium-1 molybdenum and ASTM A-302 steel.—NSA. 16944

1.6, 7.1

Gas Turbine Materials. A survey of High-Temperature Materials and Their Application to the Gas Turbine. Book, 170 pp., 1958. G. Lucas and J. F. Pollock. Available from The MacMillan Company, New York.

A survey of gas turbine materials is presented, which is intended to fill in the gap between metallurgical text books and those works concerned with the design and performance of gas turbines. The thermodynamic design of the gas turbine and the influence of fuels on the choice of materials are considered, along with a consideration of stress-temperature environment emphasizing creep. Special non-metallic and metal/non-metal combinations are discussed, and manufacturing methods are reviewed.—NSA. 16863

1.6

1957 Supplement to Book of ASTM Standards, Including Tentatives. Part 2: Non-Ferrous Metals. Book, 378 pp, 1957. Available from American Society for Testing Materials, 1916 Race Street, Philadelphia 3, Pennsylvania.

Fifty-six standards are given, covering copper and copper-base alloys, aluminum and aluminum-base alloys, magnesium and magnesium-base alloys, electrical heating materials, resistance and related alloys for wireless valves, electrodeposited metal coatings, die-cast metals, corrosion tests and general methods of testing.—MA. 15742

1.6

Materials Manual. Westinghouse Electric Corp. Atomic Energy Commission Pubn., AECU-3695, May 1957, 116 pp. Available from Office of Technical Services, Washington, D. C. Issued in looseleaf form to facilitate periodic revisions.

The considerations to be made in choosing an alloy for use under certain conditions are discussed. The properties of carbon steel, manganese-molybdenum steel, stainless steels, Stellites, Haynes-25, Inconel X, Monel, Zircaloy-2 and Zircaloy-3 are given and purchase specifications stated.—NSA. 16396

1.6, 6.4.2, 8.1.2

Aluminum in Building. E. I. Brimlow. Book, First Edition, 1957, 378 pp. Published by Macdonald & Co., Ltd., London, England.

This book provides a comprehensive store of information, based on the practical experience of builders, architects and structural engineers, related to the use of aluminum in building and allied industries. The information given is intended to enable the reader to determine where aluminum can be used to advantage and the position it should take when used in connection with other building materials. The appendices include sections on: guides to the selection of casting and wrought alloys, pretreatments for aluminum prior to painting and physical properties of high purity aluminum.—ALL. 15711

1.6, 8.4.5

Nuclear Reactor Metallurgy. Walter D. Wilkinson and William F. Murphy. Book, 388 pp. Princeton, New Jersey, D. Van Nostrand Company, Inc., 1958.

Some basic aspects of general metallurgy, including alloy theory, phase diagrams and elementary crystallography, are presented. Uranium ores, production, alloys, metallography, thermal properties, fabrication, corrosion and radiation effects are described at length. A discussion of the special problems presented by plutonium, thorium, beryllium and zirconium is presented. The use of ceramics and liquid metals in reactor technology is discussed. Metallic materials with large cross sections for thermal neutrons and the effects of neutron radiation on non-fissionable alloys are described.—NSA. 16689

1.6, 8.4.5

Nuclear Reactor Experiments. Book. J. Barton Hoag, editor. 493 pp., 1958, Princeton, D. Van Nostrand Company, Inc., Publisher.

This is a laboratory book for instruction in the field of nuclear science and engineering. It is intended to introduce scientists and engineers to the problems of designing, constructing and operating nuclear reactors. Experiments described include radiation detection, moderator and subcritical assemblies, cross sections, operating reactors, heat removal, fuel preparation, corrosion and radiation effects, separation processes and a metallurgy hot laboratory.—NSA. 16496

1.7 Organized Studies of Corrosion

1.7.1, 8.8.5, 5.9.4, 5.8.4, 5.3.4

Metal Finishing in Czechoslovakia. D. O. Bartl and O. Murdoch. Electroplating, **11,** No. 8, 263-267 (1958) August.

This article describes the work done by the Czechoslovakian Research Inst. of Corrosion and Metal Finishing since the war. Among many new developments is a cyanide zinc bath operated at 20-60 amp /sq ft. which deposits semi-bright coatings, and a large machine for zinc plating castings with an output of 700 pieces per hour. It has been found that the corrosion of zinc is much accelerated in the absence of light owing to the different corrosion products formed. A new chromate process has been developed providing a film in several possible colors. A new vapor phase inhibitor for zinc and cadmium is being tested which is said to have a wider use than inhibitors of the nitrite type. 5 illustrations.—ZDA. 16775

1.7.1, 5.8.3

The Second Conference on the Theory of Metal-Corrosion Inhibitors. (In Russian.) I. N. Putilova and S. A. Balezin. J. Phys. Chem. USSR (Zhur. Fiz. Khim.), **31,** No. 2, 522-523 (1957).

The conference was held in Moscow in January 1956. Papers were read on the work done in USSR. Putilova delivered a paper on inhibitors in nonaqueous media (fuels, oils, etc.). According to L. V. Babich, corrosion proceeds in carbon tetrachloride solution owing to the formation of hydrochloric acid. Z. A. Iofa reported the study of the influence of surface-active agents on electrochemical reactions at the solid/liquid interface. There is close connection between adsorption phenomena, electrochemical reaction, and the nature of the electrical field generated. A. T. Petrenko and L. I. Antropov showed the possibility of simultaneous action of inhibitors and cathodic polarization. In such cases the protective current can be diminished with well-chosen inhibitors 5-15 times. L. M. Romanceva reported on protection of internal-combustion engines running on sulfur-containing fuels. Good inhibitors in this case were substances retarding the oxidation of sulfurous acid to sulfuric acid.—MA. 16235

1.7.1, 6.3.17, 8.4.5

Fuel Elements Conference, Paris, November 18-23, 1957. Commissariat à l' Energie Atomique, Paris, France and Atomic Energy Commission, Washington, D. C. U. S. Atomic Energy Commission Pubn., TID-7546. Issued in two Books: (Book 1, Sessions I, II and III), 371 pp and (Book 2, Sessions IV, V, VI and VII), 448 pp.

Papers are presented in the following major categories: applied metallurgical research, natural-uranium metallic fuel elements, enriched-uranium metallic fuel elements, nonmetallic fuel elements, corrosion of uranium alloys, irradiation effects on uranium, its alloys, and its compounds, and plutonium fuel elements.—NSA. 16124

2. TESTING

2.3 Laboratory Methods and Tests

2.3.9

Determining the Decrease of Thickness of Steel Sheets and Steel Conduits Caused by Corrosion. (In French.) M. G. Pirou. Corrosion et Anticorrosion, **5,** No. 11, 339-347 (1957) Nov.

Different processes used to examine metallic walls: photography, television, acoustic, ultrasonic, radiographic, electric and magnetic methods. Application of the magnetic method to check plates and ferrous materials of average thickness, immersed or exposed to air.—BTR. 16240

2.3.9, 5.3.4

The Application of Autoradiography to Electrodeposition Studies. (In Russian.) S. M. Kochergin, G. R. Pobedimskii and A. V. Leontev. J. Phys. Chem., USSR (Zhur. Fiz. Khim.), **32,** 939-941 (1958) April.

Additions of radioactive isotope of metal being deposited were made to electrolyte and autoradiograms were obtained of radioactive deposits. Analysis of optical density curves of autoradiograms permitted determination of changes in deposit thickness with an accuracy of 0.01-0.1 micron.—INCO. 16554

2.3.9, 3.2.2, 3.7.4

Additional Information on the Interpretation of Microfractographs. (In French.) J. Plateau, G. Henry and C. Crussard. Metaux-Corrosion-Industries, **33,** No. 392, 141-162 (1958) April.

Further to earlier work, modified microfractographic technique is applied to the study of rupture mechanism and types of rupture. Preparation of replicas is described. Supporting data are given on cleavage rupture in steel (at —196 C), ductile rupture in nickel-iron alloy and 18/12/V steel, intergranular rupture in 18/12/V, nickel-chromium steels, Nicral D, 36/10 nickel-chromium steel and intergranular cracking produced during creep in alloy of Nimonic 80 type.—INCO. 16713

2.3.9, 5.9.4

Oscillographic Method of Testing Aluminum Oxide Surface Coatings. (In German.) G. Hahn. Metalloberfläche, **12,** No. 6, 170 (1958) June.

An oscillographic method of detecting discontinuities and flaws in anodic oxide coatings on aluminum is described. With this technique no superimposed voltage

is required and it is possible to determine characteristics of the oxide film with smaller variations and errors in the results than one usually encounters when using conventional electric indicators.—ALL. 16490

2.3.9, 3.8.4

The Diffraction of Electrons by Thin Films. Final Technical Report. S. H. Bauer. Cornell Univ., Dept. of Chemistry. U. S. Atomic Energy Commission Pubn., Contract Nonr-401(06), Feb. 1, 1958, 221 pp. Available from Cornell University, Ithaca, New York.

A series of electron diffraction studies, both experimental and theoretical, is presented. Section A is devoted to a brief description of apparatus used in the experiments. In section B the development of quantitative methods for estimating intensities in diffraction patterns produced by 50-kv electrons impinging on thin films and on the outer layers of crystallites is described. In section C the radial distribution method is discussed and empirically tested. Section D is an abstract of a thesis on the precision determination of lattice parameters of polycrystalline specimens giving broad diffraction peaks. Section E is a literature survey of theoretical and experimental work on surface energies of microcrystallites and of the distortions in the outer layers which accompany such surface effects. Section F covers the experimental studies of collodion films. Section G is a theoretical analysis of the electron diffraction shape factor for a thin film. Section H consists of two treatments of the vexing and difficult problem of plural and multiple electron scattering by thin films and microcrystallites. Section J is an abstract of a thesis on the measurement of contact potentials of freshly evaporated films of silver, copper and aluminum and changes in these films due to adsorption of hydrogen and oxygen.—NSA. 16453

2.3.9, 3.5.7

New Ultrasonic Method of Measuring Compressive Forces. H. Krachter. Werkstatt u. Betrieb., **91,** No. 5, 246-248 (1958); Engrs. Digest., **19,** No. 6, 223-224 (1958) June.

Ultrasonic method described permits measurements of compressive forces by simple means, with remote indication of results. Over range of pressures from 200-2000 kg/cm², accuracy is ± 1.5%. With suitable electrical instruments, accuracy can be even better with 1.5%. Experiments with stainless steel plates are described. Diagrams, graphs.—INCO. 16840

2.3.9, 8.4.5

Advances in the Application of Radioisotopes and Nuclear Irradiation in Metallurgical Research. M. T. Simnad. General Dynamics Corp. U. S. Atomic Energy Commission Pubn., A/CONF. 15 P/803, 21 pp. Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958. Available from Office of Technical Services, Washington, D. C.

A wide range of topics is covered since radioisotopes have found application in almost all branches of metal research, namely, in studies of segregation, friction, vapor pressure, slag-metal reactions, electrolytic action and corrosion on metal surfaces, diffusion and oxidation. A description is given of recent unpublished work on heterogeneous nucleation of metals from the vapor phase. Radioisotopes have provided a unique means for studying the metal condensation process from atomic beams on various substrates. (auth)—NSA. 16845

2.4 Instrumentation

2.4.2, 2.3.9, 5.9.3

Electromagnetic Test Measures Effects of Shot Peening. P. M. Unterweiser. Iron Age, **181,** No. 23, 121-123 (1958) June 5.

Two Russian scientists, N. A. Petrova and M. Y. Shashin, have experimented with a non-destructive method that determines depth of compressed layer and also measures quality of shot peening. Their technique is based on changes in electrical and magnetic properties that result from plastic deformation found in compressed layer. It can be applied to magnetic materials only. Advantages of electromagnetic measurement are that it can be applied to both hard and soft steels, it can be used as a routine method to check shot-peening quality once calibration curves correlating thickness of the strain-hardened layer with readings of instrument are available, and it can determine optimum peening conditions for a given part before part is put into production. Table shows depths of compressed layer of three bar sizes of a chromium-nickel-molybdenum-vanadium steel. Graphs.—INCO. 16881

2.4.3, 6.4.2, 3.5.8

Early Detection of Fatigue in Aluminum Alloys by Ultrasonics. K. Sittel, M. Herman and R. C. Good, Jr. The Franklin Inst. for U. S. Wright Air

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Development Center, U. S. Air Force. May, 1958, 45 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 151019).

A literature survey was conducted to determine the use of ultrasonics for early detection of fatigue damage in metals, specifically in aluminum used as an aircraft structural material. Present acoustical methods are described and their applications in fatigue studies are given. Effects which occur during early stages in the fatigue life of a specimen are described and advantages are given for using ultrasonic waves of frequencies in the megacycle range. Also discussed are the possibilities of using the attenuation-temperature relationship for detecting dislocation motion and of apply ultrasonic waves to incipient fatigue detection. Experimental work included design and construction of a resonant-type fatiguing machine and an electro-acoustic driving system for ultrasonic waves.—OTS. 16851

3. CHARACTERISTIC CORROSION PHENOMENA

3.2 Forms

3.2.2

Hydrogen in Steel, with Special Reference to Flakes and Cracks Formed at Elevated Temperatures. V. Koselev. Neue Hutte, **2,** No. 10, 601-604 (1957).

Study of primary and secondary causes of formation of flakes and segregation-induced internal cracks in steel. Connection between pressure in steel and residual stresses in conjunction with gamma-to-alpha transformation temperature is discussed. Well-documented study of flakes and internal cracks in a rotor forging is given. Mechanism and time of formation of flakes and internal cracks are described. Photomicrographs. —INCO. 16218

3.2.2, 3.7.3

A New Concert of Hydrogen Embrittlement in Steel. J. G. Morlet, H. H. Johnson and A. R. Troiano. Case Institute of Technology for U. S. Wright Air Development Center, U. S. Air Force. March, 1957, 50 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington, D. C. (Order PB 131200.)

Current theories of hydrogen embrittlement were critically evaluated from an investigation of the influence of plastic strain on the aging characteristics of hydrogenated 4340 steel. It was observed that the recovery curve is strongly modified by plastic deformation either before or after hydrogenation, a modification not explained by current theories. The study demonstrates that embrittlement results from hydrogen in solution, and that hydrogen in internal voids is non-damaging. Voids created by plastic strain occlude hydrogen from the metal surrounding the void. This process is accompanied by a ductility increase, indicating that the hydrogen in the voids is non-embrittling. Aging curves for specimens hydrogenated after cold working were consistent with this analysis. Embrittlement decreases with increasing prior strain, since less hydrogen is left in the lattice as a result of the increased void capacity.—OTS. 16249

3.2.2, 7.1, 3.5.8, 4.3.3

Investigation of Corrosion Cracking of Steam Turbine Disks. V. M. Nikiforova and N. A. Reshetkina. Energomashinostroenie, **3,** No. 10, 19-22 (1957) Oct. (In Russian.)

Shows that the basic cause of disk fracture is corrosion cracking resulting from simultaneous influence of alkali salts and high working stresses in the metal.—BTR. 15472

3.2.2, 3.4.8, 4.3.2, 6.2.3

Contribution to the Study of Factors Governing Rupture of Mild Steel Subjected to Tension in a Hydrogen Sulphide Saturated Acid Solution. (In French.) Eugene Herzog. Comptes Rendus, **244,** 1499-1502 (1957) March 11.

Importance of the nature of carbides on the propagation of ruptures in an acid medium, whether saturated with hydrogen sulfide or not.—MR. 16180

3.4 Chemical Effects

3.4.3, 6.4.2

Technique of Aluminum Corrosion Measurement. A. Russell. General Electric Co., Hanford Atomic Products Operation. U. S. Atomic Energy Commission Pubn., HW-49631, March 4, 1957, 10 pp. Available from Office of Technical Services, Washington, D. C.

Methods of measuring the corrosion product formed on aluminum and various aluminum alloys are discussed. The aluminum was in the form of wafers or coupons measuring approximately 1 x 1 x 0.1 in. The coupons were exposed to high temperature water (above 200 C) in the presence of injected phosphoric acid for various time intervals. X-ray defraction analysis of the corrosion product has shown it to have the formula 2 $Al_2PO_4(OH)_3$ or $(Al_2O_3)_2$. $P_2O_5 \cdot 3H_2O$. There are several general methods of measuring the amount of corrosion product included (auth).—NSA. 16304

3.4.3, 2.1.2

Diagnosis of the Causes of Corrosion by Means of Analyzing Corrosion Products. (In German.) Gerhard Schikorr. Werkstoffe u. Korrosion, **9,** No. 8/9, 552-556 (1958) August-September.

A comprehensive survey of methods of using the analysis of corrosion products as the basis for establishing the diagnosis for the underlying causes, is presented. Accordingly, the chemical composition, distribution, color, rate of dissolution and crystal structure of the corrosion products are studied and the probability of error is evaluated. Dr. Schikorr's article is followed by a complementary note by W. Feitknecht.—ALL. 16858

3.4.3, 3.4.7, 3.5.4

Van De Graaff Study: Second Interim Report on the Deposition of Corrosion Products Under Irradiation. G. P. Simon, B. G. Schultz and Y. Solomon. Bettis Technical Review, **1958,** 43-51, March; U. S. Atomic Energy Commission Pubn. WAPD-BT-7.

For any pH studied, deposition of soluble or colloidal iron "species" on Zircaloy-2 specimens at 450 F and with less than 0.1 ppm oxygen in the water is about 26% of the material passing the target with radiation and 6% without radiation. Under these same conditions of temperature and oxygen concentration, the amount of corrosion product deposited on the Zircaloy-2 specimens varies inversely with the pH. This results from the fact that the release rate of corrosion products varies. The efficiency of deposition for soluble or colloidal iron species appears to remain unchanged with varying hydroxyl ion concentration. Graph, tables, diagrams. —INCO. 16914

3.4.9, 3.4.6, 8.4.5, 5.3.4

The Effect of Oxygenated Water on Clad-and-Defected UO_2 Fuel Specimens. J. M. Lojek and W. T. Lindsay, Jr. Bettis Technical Rev. 33-42 (1958) March; U. S. Atomic Energy Comm., Pubn. WAPD-BT-7.

Out-of-pile tests were run to determine extent of deterioration and contamination resulting from effect of oxygenated water on clad, defected Pressurized Water Reactor fuel elements. Small pits in fuel pellets and some discoloration in cladding resulted, but weight loss was small. It was concluded that even defected cladding offers protection sufficient to avoid appreciable harm to rods, pellets or primary system. Photomicrographs, photos, diagrams.—INCO. 16924

3.5 Physical and Mechanical Effects

3.5.8, 6.3.6

Stress-Corrosion of Brass. (In Polish.) C. Niewiadomski. Rudy i Metale Niezelazne, **2,** No. 1, 13-20 (1957).

Deals with the causes, mechanism and prevention of season cracking. Among the causes of cracking are internal stresses in the presence of zinc in the lattice, abnormal grain-size and the type of storage atmosphere. Usually several of these combine to produce stress-corrosion. Cracking is best detected by exposure to 25 or 5% ammonium hydroxide solution. The mercuric chloride test is not entirely satisfactory, for it reveals only intercrystalline cracking. The mechanism of cracking is not fully known but probably involves the chemical combination of zinc and ammonia. Cracking can be minimized or eliminated by uniform plastic deformation, stress-relief (250-280 C in forced-air circulation) followed by rapid cooling, surface conditioning (mechanical stress-relieving), protective coating and suitable storage conditions. 35 references.—MA. 15867

3.5.8, 5.3.4

Factors Affecting Residual Stress in Electrodeposited Metals. A critical Evaluation. J. B. Kushner. Electroplating School. Metal Finishing, **56,** Nos. 4, 5, 6, 7; 46-51; 82-87; 56-60; 52-55, 59 (1958) April, May, June, July.

Purpose of study was to obtain more definite relationships between residual stress and causative factors, as determined under standard set of operating conditions, and to examine these results and those of other workers in the field. Hydrogen and hydride theories, excess energy theories and occluded matter theories are reviewed. Metal used in experiments was nickel which is subject to large variations in stress. Residual stress was measured by Stresometer developed by author. Relationship between residual stress and deposit thickness, effect of cathode film, effect of temperature, effect of current density and effect of contaminants and addition agents are discussed. In considering role of metal surface, surface cleanliness and adhesion, surface finish and residual stress, effect of grain size and nature of substrate metal are covered. Stress after effect and hydrogen uptake are discussed and suggested theory for cause

of residual stress is summarized. Data are presented for nickel deposits from sulfamate, fluoborate, bromide, sulfate, chloride, iodide and nitrate baths. Numerous tables, graphs, 63 references.—INCO. 16817

3.5.8, 3.8.4, 6.4.2

A Contribution to the Theory of Stress Corrosion in Aluminum-4% Copper Alloys. William H. Colner and Howard T. Francis. Armour Res. Foundation, Illinois Institute of Technology. J. Electrochem. Soc., **105,** No. 7, 377-384 (1958) July.

Stress cracking of aluminum-4% copper alloys was found to be greatly influenced by exposed area of the specimen (small areas showed long time-to-failure). Experiments with grain boundary-grain center couples showed that neither potentials nor couple current were influenced sufficiently by stress to account for the acceleration of failure by stress. At constant applied anodic current, however, stress was found to direct the attack perpendicular to applied stress. The theory of Champion and Logan is substantiated. A refinement is suggested in which the last stages of failure result from direct and rapid local attack on film-free metal. 16029

3.5.8, 4.6.11, 7.1

Fatigue Performance of Marine Shafting—Laboratory and Service Tests. T. W. Bunyan. Paper before Am. Soc. for Testing Materials, Symposium on Large Fatigue Testing Machines and Their Results, 60th Annual Mtg., Atlantic City, June 18, 1957. ASTM Special Technical Pubn. No. 216, 59-79; disc. 79-80 (1958).

Comparison of laboratory and service tests. Of total failures in marine shafting, about 80% are associated with corrosion fatigue due to ingress of sea water to large end of tailshaft cone. Operational and constructional stress raisers are discussed and include corrosion pitting at points of stress concentration caused by contamination of lubricating oil by salt water on corrosive products of combustion. Photos, diagrams.—INCO. 15605

3.5.8, 3.2.3, 6.3.6

Mechanical Stresses During Formation of Oxide Layers. Pt. I. Copper Oxidation. (In German.) Walther Jaenicke and Siegfried Leistikow. Z. Physik. Chem., (Neue Floge), **15,** Nos. 1-6, 175-195 (1958) April.

Causes of mechanical stresses in the oxide layer as well as in the metal itself.—BTR. 16076

3.5.8, 3.5.9

Characteristics of Fatigue Failure of Alloys at High Temperature. V. A. Parfenov. Metalloved. I Obrabotka Metal., No. 2, 19-22 (1958) Feb. Translation available from Henry Brutcher, Technical Translations, P. O. Box 157, Altadena, California.

Scarcity of literature data on high-temperature fatigue of creep-resisting alloys. Variation of fatigue limit with temperature. Possibility of studying the structural changes during fatigue testing by micrographic analysis. Results. Comparison of fatigue strength with rupture strength curves; significance of ratio of fatigue strength to rupture strength in assessing the quality of high-temperature alloys. Relative probability of failure of alloys above a certain temperature owing to cyclic as against static stressing, demonstrated on the basis of a nickel-20% chromium-2½% titanium alloy. 7 figures.—HB. 15695

3.5.8, 3.8.4

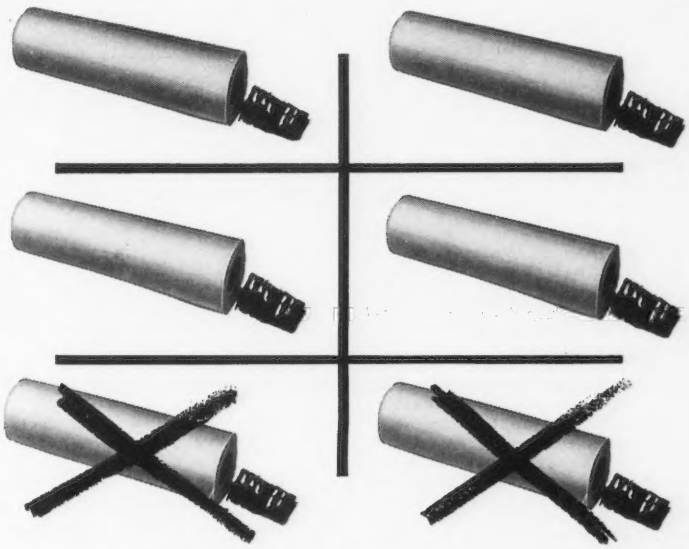
The Influence of Surface Layers on Stress-Corrosion in Steel. H. Gräfen. Archiv. Eisenhüttenw., **29,** 225-229 (1958) April.

Investigation, using apparatus specially developed by author, of stress-corrosion in steels, with particular emphasis on role of surface layers, such as oxide films. Supporting data are given on plain, low-alloy nickel-chromium-molybdenum, and austenitic steels exposed to solutions containing potassium dichromate, nitrate and halide additions.—INCO. 16034

3.5.8, 3.7.3, 6.3.15

Stress Relief, Annealing and Reactions with Atmosphere of Titanium and Titanium Alloys. D. J. Maykuth. Titanium Conference, **1957,** 44 pp. Available from the American Society of Metals, 7301 Euclid Avenue, Cleveland 3, Ohio.

The origin of residual stresses is reviewed, and some of the most commonly observed effects of such stresses in titanium and its alloys are described. The principles of heat-treatment are discussed, appropriate stress-relief and annealing treatments are tabulated for various titanium bar and sheet alloys. The effects of time and temperature on air contamination of titanium and methods of measuring and controlling surface contamination are described. The sources of hydrogen causing contamina-



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tion of titanium are described and the effects of hydrogen on titanium discussed. Methods of controlling hydrogen content are outlined. 26 references. —MA. 15986

3.5.8, 6.3.6

Season Cracking of Brass. (In French.) J. M. Pouvreau. Cuivre, Laitons, Alliages, **36,** 41-45 (1957) March-April.

Though the causes and mechanisms of season cracking, or, more correctly, stress-corrosion cracking, of brass are not yet fully understood, predominant factors are the chemical composition of the alloy, the existence of internal or external stresses, corrosive atmospheres, sudden temperature changes, and the form of the brass products. Various aspects of these factors are discussed, and examples of the phenomenon illustrated. —MA. 15890

3.5.8, 6.4.2

Stress Corrosion of Aluminum Alloys. (In French.) F. A. Champion. Corrosion et Anticorrosion, **6,** No. 4, 132-140 (1958) April.

Definition of stress corrosion. Rupture mechanism. Types of stresses used. Aluminum alloys without true sensitivity to stress corrosion. Laboratory tests and outside tests. Protective measures.—BTR. 16042

3.5.8, 6.4.2, 1.3

Stress Corrosion of Wrought Aluminum Alloys. (In German.) H. Vosskühler. Korrosion IX, 45-62 (1958). Special Publication of the Deutsche Gesellschaft für Metallkunde, Frankfurt am Main, Germany.

Review and evaluation of research work carried out since 1944 on the stress corrosion effect in wrought aluminum alloys. Insofar as the mechanism of stress corrosion is concerned, there is no difference in behavior between homogeneous and heterogeneous alloys. Basic condition of the development of stress corrosion is the presence of potential differences in the material. It is of little or no importance whether these potential differences are caused by the dissimilarity of structural components or by various physical factors. In the case of a substantially heterogeneous alloy only the precipitation at the grain boundaries is operative (intercrystalline stress corrosion); in the case of alloys which are substantially homogeneous but electrochemically heterogeneous, besides the grain boundaries, any plant perpendicular to direction of tensile stress may become anodic. Under such conditions stress corrosion develops in the transcrystalline way. The evolution of the process passes through two stages: (1) localized attack whereby the mechanical equilibrium being disturbed, plastic flow (2) of the material sets in which disrupts the oxide film and exposes bare metal to a further, accelerated attack. A bibliography is presented.—ALL. 16009

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3.6 Electrochemical Effects

3.6.5, 3.8.2

The Electrochemical Series of Metals in the Eutectic Melt of Sodium Fluoride-Potassium Fluoride. Kai Grjotheim. Z. physikal. Chem., Frankfort, **11,** No. 3/4, 150-164 (1957). (In English.)

A cell is described consisting of two platinum crucibles forming half-electrodes connected by a salt bridge. The crucibles carry molten sodium fluoride-potassium fluoride eutectic and electrodes. For aluminum electrode measurements a submerged alumina crucible is used. Voltage measurements are carried out at 850 C under dry inert gas. With nickel as zero the following electrochemical series under these conditions was found to be. Al^{III}—1.5, Mn^{II}—104, Cr^{III}—0.70, Fe^{III}—0.12, Co^{II}—0.07, $Ni^{II}O$, Cu^{I} + 0.48, Ag^{I} + 0.64. 13 references.—MA. 16201

3.6.5, 4.3.2

On the Electrochemical Behavior of Chromium and Tungsten Carbides. (In Japanese.) Saburo Shimodaira and Yoshinobu Sawada. J. Japan Inst. Metals (Nippon Kinzoku Gakkai-Si), **21,** No. 4, 271-275 (1957).

Current density/electrode potential curves are given with 1 and 10% hydrochloric acid solutions, and with 1 and 18% sulfuric acid solutions. Tungsten carbide is less stable than chromic carbide to the oxidizing and reducing actions of acids.—MA. 16336

3.6.5, 4.3.2, 6.3.6

Maximum Cathode Current Density and the Rate of Corrosion of Copper in Nitric Acid Solutions. (In Russian.) I. V. Oknin. J. Applied Chem., USSR (Zhur. Priklad. Khim.), **30,** No. 9, 1330-1339 (1957) Sept.

Comparison of the maximum cathode current density, measured on a flat platinum electrode, and the rate of copper corrosion in nitric acid solutions at 25, 60 and 90 C.—BTR. 16260

3.6.5, 3.8.2, 6.3.8

Electrolytic Corrosion of Lead in Dilute Sodium Hydroxide and Sulfuric Acid Stolutions. (In Japanese.) M. Tagaya and M. Hori. J. Japan Inst. Metals, **21,** No. 9, 558-561 (1957) September.

In an alkaline solution (pH 11.3, 0.008% sodium hydroxide) severe anodic corrosion of lead suddenly begins at 160 mV nobler than the natural potential of lead in the same solution. The current density at this point is 2 mA/dm². In dilute sulfuric acid solutions (pH 1.3, 2.8) anodic corrosion slowly occurs in the range from 100 to 2000 mV nobler than the natural potential in these acid solutions on account of lead sulfate coating. The flow-out current density in this range is 10 mA/dm² or less. In the next step lead dioxide is formed on the anodic surface and anodic corrosion decreases on account of oxygen evolution at the current density of 20-80 mA/dm². For the cathodic protection of lead in these alkaline and sulfuric acid solutions, the optimum electrode potential and the flow-in current density are —200 mV and 2-6 mA/dm². Overprotection exceeding this limit may cause cathodic corrosion of lead by accumulated alkali.—JSPS. 16425

3.6.6, 6.4.2, 5.4.2, 8.1.2

The Behavior of Aluminum in Contact with Cement Concretes; Present Day Status of the Pertinent Knowledge. (In Italian.) A. Prati. Alluminio, **27,** No. 9, 381-385 (1958) Sept.

Values of material removal in aluminum and various aluminum alloys caused by the presence of cement concretes of various types, are plotted against time. The findings indicate that in such structural and architectural uses it is necessary to protect the metal with coatings which are impervious to moisture and electrically insulating. Bituminous and asphalt materials are considered to be most satisfactory. For reasons of preserving the good appearance of the building metacrylic resins are used with good success.—ALL. 16928

3.6.8

The Effect of Polarization on the Mechanical Properties of Steel. (In Russian.) G. V. Karpenko and R. I. Kripyakevich. Acad. Sci., Ukranian SSSR, Inst. of Mechanization and Automation (Doklady Akad. Nauk SSSR), **120,** 827-829 (1958) June 1.

Effects of polarization on the mechanical properties of steel were tested in various electrolytes with anode and cathode polarization with external current sources.—NSA. 16832

3.7 Metallurgical Effects

3.7.3, 1.7.1

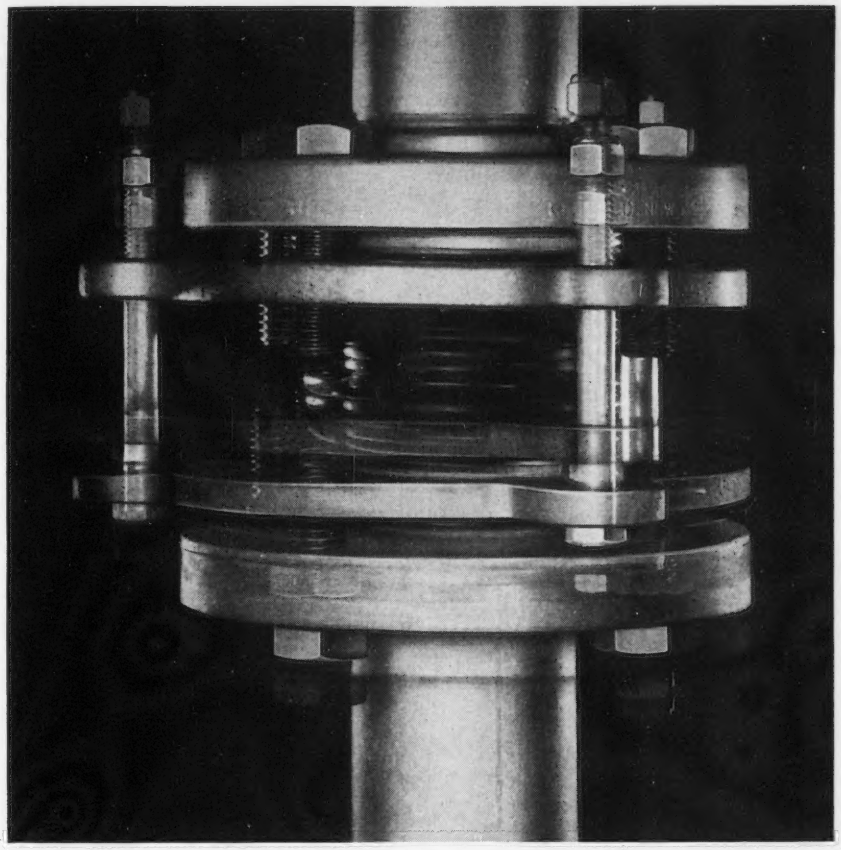
Atomic Energy Commission Welding Conference. Minutes—AEC Welding Committee Meeting, Argonne National Laboratory, November 9 and 10, 1953. Sept. 30, 1954. Division of Reactor Development, AEC. U. S. Atomic Energy Commission Pubn., WASH-181 (Del.), Declassified with Deletions March 10, 1957, 372 pp. Available from Office of Technical Services, Washington, D. C.

The principal problems discussed concerned the inert arc welding of stainless steel in which nitrogen was used as a purging gas, the corrosion of welds and the properties of brazing alloys used in the fabrication of metallic plate type fuel elements.—NSA. 15738

3.7.3, 5.3.4

Maintaining the Solderability of Silver-Plated Parts. A. Keil. Metalloberflache, **11,** No. 10, 334-335 (1957).

When silver-plated parts are stored they tend to tarnish; if they are for electrical-engineering purposes, any subsequent soldering operations become difficult, as the silver sulfide film formed is not removed by the resin flux generally employed. This difficulty can be avoided by a thin flash of gold, a thin



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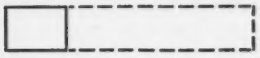

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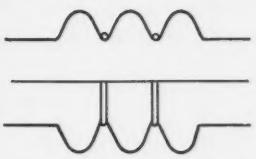

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film of polyurethane resin or a chromate passivation-treatment; paraffin wax or lacquer coatings are unsatisfactory.—MA. 15900

3.7.3, 5.4.2, 6.2.5

Problems in Simultaneous Heat-Hardening and Ceramic Coating of No. 420 Stainless Steel. E. L. Bradley. Ferro Student Award Paper before Am. Ceram. Soc., 59th Ann. Mtg., Dallas, May 5-9, 1957. Am. Ceram. Soc. Bull., **37**, No. 5, 222-226 (1958) May.

Purpose of investigation was to protect Type 420 from scaling during its high temperature heat treatment, and to impart to the metal a ceramic coating which would be white, nonporous, flexible, resistant to thermal shock and have low neutron absorption cross section. It is shown that cooling contraction curve for Type 420 can be useful in determining optimum softening point which its enamel coating should possess. Glass with low linear coefficient of thermal expansion and low softening point will not produce satisfactory coating for subject metal. Thermal expansion curves are shown for Type 420 and Pyrex.—INCO. 16045

3.7.3, 6.4.2

The Metallurgy of Welding Aluminium and Its Alloys. W. I. Pumphrey and E. G. West. Brit. Welding J., **4**, No. 7, 297-306 (1957).

Problems initially encountered in the welding of aluminum alloys are reviewed. The effects of temperature, oxygen, hydrogen, other elements in the parent metal and the weld and of post-welding treatment, on the physical, mechanical and corrosion properties are discussed. Pumphrey and West deal mainly with fusion welding, but brief reference is made to resistance welding, especially spot and pressure welding.—MA. 15914

3.7.3, 6.4.4

The Metallurgical Background to Magnesium Alloy Welding. E. F. Emley. Brit. Welding J., **4**, No. 7, 307-321 (1957).

The relevant properties of commercial magnesium alloys are reviewed and the ease with which they can be fusion-welded is considered on the basis of general observation, data in the literature and test results obtained with Houldcroft's welding jig. Resistance welding, the repair of castings and stress relief are mentioned. Methods available for welding magnesium are reviewed, and special problems arising from flux entrapment in gas welding and with zirconium-bearing alloys, which may be susceptible to corrosion in gas-welded seams, are discussed. 42 references.—MA. 15769

3.7.3, 3.7.4, 6.3.10

On Directionality of High Nickel-Chromium Weld Metals. K. Watanabe. Paper before Japan Weld. Soc., Spring Mtg., 1956. J. Japan Weld. Soc., **27**, No. 1, 18-25 (1958) Jan.

The directionality of crystal grain of high nickel-chromium weld metal with particular reference to Inconel was investigated in comparison with that of 25 chromium-20 nickel and 18 chromium-8 nickel steels by checking their tensile property, impact value and microstructure. Mechanical property of Inconel weld deposit varies considerably according to the welding direction. It gives the lowest value along the progressive direction of welding and the highest value toward the piling up directions where the growth of crystal grains take place. As nickel content of welding rod increases, the length of a columner grain increases and the grains themselves tend to be coarsened, giving a remarkable directionality toward the welding direction. When the base metal is preheated before welding, the grains of the welded Inconel will be coarsened and the coarsened grains may deteriorate the mechanical property. But these defects can be remedied by a rapid cooling after welding. Precipitation of niobium nitrides or carbides along the grain boundaries is the main cause of cracking or of deteriaration of the mechanical property. The addition of manganese to the Inconel weld metal will prevent the above precipitation, although it may cause grain growth to some extent. The welding rods thus prepared give no crackings giving a superior mechanical property to the welded joint.—INCO. 16941

3.7.3, 8.4.5

Special Welding Problems in Nuclear Engineering. A. Luthy. Brown Boveri Review, **45**, No. 6, 272-279 (1958) June.

Deals briefly with normal welds in pressure vessels and pipes, then examines some special cases in nuclear engineering where execution of pressure-tight welds presented certain problems. In heavy-water heat exchanger, outer casing of boiler plate, inner part carrying nest of tubes and tubes themselves are of 18-8. Unit differs from conventional design by having second tube-

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plate situated few centimeters from first to create safety chambers. Ends of 18-8 tubes for low pressure are rolled into tube plates; for high pressure they are rolled in and welded by Argonarc process with non-consumable electrode. Electrode is tungsten containing 0.25-0.30 zirconium. In heavy-water pump, pressure-tight welds in very thin (0.5 mm) sheets of 18-8 are produced with nickel-chromium-manganese filler metal, a strip of which is placed between edges of contact surfaces. Alloy melts at 1071 C at which temperature it slightly wets metal. Use of this filler metal increases depth of weld cross-section and renders it completely pressure-tight. Same type of edge joint is applied to weld non-alloy steel to nickel-cobalt steel for object exposed to high vacuum pressure. Considerable use is made of clad steel sheet. Macrograph shows seam in sheet containing 20 chromium, clad with 16/13 stainless steel. For first pass, type 22/4 electrode is used for ferritic side, and second pass on austenitic side is made with 16-13 electrode. Some parts are clad by weld surfacing; metal inert gas process is used to apply 18/9 layer on boiler plate.—INCO. 16917

3.7.4, 3.7.3, 6.2.5

Effect of Columbium on the Structure of Welds in Austenitic 18-8 Steels. (In Russian.) N. F. Lashko. Avtomat. Svarka, **11,** No. 6, 84-87 (1958). Translation available from Henry Brutcher, Technical Translations, P. O. Box 157, Altadena, Calif.

Particulars on the phases which may actually be present in columbium-stabilized 18-8 steel. Refutation of occurrence of the new phases claimed by W. O. Binder and also the H-phase claimed by Mukhin and Pal'chuck. Proof of the real nature of these phases, based on X-ray data. Table.—HB. 16830

3.7.4, 3.8.4, 6.3.6

Grain-Growth in α-Brasses. P. Feltham and G. P. Copely. Acta Metallurgica, **6,** No. 8, 539-542 (1958) August.

In alpha-brasses dependence of mean grain diameter D on time of isothermal annealing is expressed in equation which differs from that for pure metals by an additional term embodying parameters C_{Zn} representing zinc concentration, H', an activation energy equivalent to 9L where L is latent heat of melting, and T_s, which is numerically equal to solidus temperature of typical 70/30 alpha-brass. Magnitudes of H_o (20.2 kcal/gram atom) and K_o (3×10^{-3} cm^2/sec) are such as would be expected for unalloyed copper. Form of concentration-dependent term suggests that atom transfer across grain boundaries is facilitated by relaxation of zinc-copper bonds which is energetically equivalent to melting to group of 9 atoms, 9 being assumed to be co-ordination number prevalent at grain interfaces. Graphs.—INCO. 16819

3.7.4, 6.3.6, 3.7.3

The Martensite Transformation of the Beta Phase in Copper-Aluminum-Nickel Alloys. D. Hull and R. D. Garwood. J. Inst. Metals, **86,** Pt 11, 485-492 (1958) July.

Two types of martensite transformation of the metastable beta-phase were observed in an alloy containing 79.5 copper, 12.8 aluminum and 7.7 nickel. When the quenched alloy is cooled to 0 C, the plates formed are relatively fine and free from substructure (β), but on tempering the quenched alloy and subsequent cooling to room temperature, a coarse-grained product (σ'), with a distinct secondary structure, is formed. Both types of martensite form isothermally when the alloy is aged at room temperature after a suitable heat-treatment. This fact and the conditioning of the beta transformation into σ' martensite, which occurs during tempering, are thought to be due to the growth of the ordered domains of the Cu_3Al superlattice. Photomicrographs, graphs.—INCO. 16821

3.7.4, 3.8.4

Some Magnetic and Crystallographic Properties of the System $Li_x^+Ni_{1-2x}^{++}Ni_x^{+++}O$. J. B. Goodenough, D. G. Wickham and W. J. Croft. Massachusetts Inst. of Technology. J. Phys. Chem. Solids, **5,** No. 1/2, 107-116 (1958).

Crystallographic and magnetic measurements of system $Li_x^+Ni_{1-2x}^{++}Ni_x^{+++}O$ for entire range $O \leqslant x \leqslant 0.5$ are reported. This system is formed when nickel monoxide and lithium oxide are heated together at 1200 C in the air. For values of $x \leqslant 0.3$, lattice appears to be antiferromagnetic and is cubic above Curie temperature. For composition range $0.3 < x < 0.5$, lattice is rhombohedral and ferrimagnetic, maximum magnetic moment per gram of 31 e.g.s. cm^3/g being observed at $x = 0.46$. Maximum ferrimagnetic Curie temperature of 241 K was observed for composition $x = 0.41$. These properties are interpreted in terms of partial ordering of lithium and nickel ions into alternate sets of (111) planes which couple antiparallel to one another, moment within any set being either ferromagnetically coupled or paramagnetic, depending on

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number of non-magnetic near neighbors in neighboring planes. Lattice parameters are tabulated.—INCO. 16815

3.7.4, 6.3.6

The Development of Microscopic Inhomogeneities of Deformation in Polycrystalline 70:30 Brass: Some Effects of Method of Deformation. L. E. Samuels and M. Hatherly. J. Inst. Metals, **86,** Pt. 10, 442-446 (1958) June.

Incidence of 2 types of microscopic inhomogeneity of deformation (lamellar slip bands and kink bands) in polycrystalline 70:30 brass was found to vary markedly with different methods of unidirectional deformation. Evidence was obtained that the kink-band type of inhomogeneity does not influence strain-hardening, but that the slip bands do have a small influence, particularly when the bands develop on multiple systems. This is interpreted as indicating that strain-hardening in this material is due largely to interference between intersecting slip systems. Graphs, photomicrographs, 18 references.—INCO. 16866

3.7.4, 3.7.3, 6.2.3, 6.2.4

Investigations on the Banded Structure in Plain Carbon and Alloy Structural Steels. (In German.) E. Plöckinger and A. Randak. Stahl and Eisen, **78,** No. 15, 1041-1054; disc. 1054-1058 (1958) July 24.

Investigation of causes of non-uniform structure of structural steels and microscopic determination of transformation structure of plain carbon and low alloy case-hardening steels and steels suitable to quenching and subsequent drawing (e.g. Ck 35, 34 Cr 4, 16 CrMo 4, 25 CrMo 4 and 34 CrMo 4), in as-cast or hot-worked conditions, after different rates of cooling. Effect of prior diffusion annealing and of repeated additional hot-working is considered. Phenomena leading to banded structure are discussed. Effect of alloying elements (silicon, manganese, phosphorus, chromium, molybdenum and nickel) is investigated. Nickel is found to lower the A_3 transformation point. Tables, graphs, numerous photomicrographs.—INCO. 16965

3.7.4

An Investigation of the Phase Equilibrium Diagram in the Cobalt-Chromium-Titanium System. (In Russian.) B. G. Livshits and Ya Khorin. Zhur. Neorg. Khim, **3,** No. 3, 685-693 (1958).

Investigation to study the cobalt corner of the cobalt-chromium-titanium phase equilibrium system containing up to 55 chromium and up to 45 titanium. Tables, graphs, photomicrographs.—INCO. 16948

3.7.4, 3.7.3

Temporary Changes in Mechanical Properties (of Steel) During Phase Transformations. (In Russian.) V. G. Vorob'ev. Metalloved. I Obrabotka Metallov., No. 3, 35-38 (1958) March. Translation Available from Henry Brutcher, Technical Translations, P. O. Box 157, Altadena, California.

Literature data on uncommonly high plasticity of zinc alloy, copper alloys and various machinery and tool steels while undergoing phase transformations. Author's experiments aimed at amplifying Bochvar's theory on 'superplasticity' as due to transfer of atoms by diffusion (solution-precipitation) in interphase regions. Determination of plasticity by measuring deflections of various steel specimens under load during the austenite-to-martensite transformation at subzero temperatures (where diffusion plays practically no role). Evaluation of results and their usefulness in developing new (distortion-free) heat treating practices. 3 figures.—HB. 16849

3.7.4, 3.7.3, 6.3.13

Recrystallation Diagram of Tantalum. (In Russian.) E. M. Savitskii et al. Proc. Acad. Sci., USSR (Doklady Akad. Nauk SSSR), **118,** No. 4, 720-722 (1958). Translation available from Henry Brutcher, Technical Translations, P. O. Box 157, Altadena, California.

Usefulness of recrystallation diagram of tantalum (relating grain size to amount of reduction and temperature of subsequent anneal) for selection of an annealing practice giving the best possible mechanical properties. Specimen preparation. Results of micrographic and X-ray studies. Prerequisite to high ductility after anneal at about 1600 C (2910 F). Recommended annealing temperatures for tantalum metal with a fine grain and good mechanical properties. Author's recrystallation diagram. 3 figures.—HB. 16852

3.8 Miscellaneous Principles

3.8.3, 6.3.10, 3.4.8

The Anodic Behavior of Nickel. Pt. I. Effect of Components of Electrolyte. Prof. Nelson F. Murphy and Bharat C. Oza. Bull. of the Virginia Polytechnic Inst., Engineering Experiment Station Series, No. 125, **51,** No. 7, 3-18 (1958) May.

This is an investigation of the quantitative effects of the constituents of the electrolyte on the behavior of three nickel anodes. One anode was a portion of an electroplating anode rolled and depolarized. The second was a piece of sheet nickel containing less than 0.02% carbon. The third was a sheet anode of A-A, commercial grade nickel, containing 0.05% carbon and 0.5% cobalt.

The anode potential and current density at which nickel became passive was found to be a reproducible value that increased as the temperature increased. The well known effect of the chloride ion in destroying the passivity of nickel was studied quantitatively. The presence of coexisting sulfate and chloride ions was shown to counteract the effect of nickel ions. The limiting current density was increased by the presence of sulfate ion, the magnitude of the increase depending on the concentration of nickel in the solution. Boric acid did not affect the limiting current density very much at low concentrations of nickel ions, but had an appreciable effect at higher nickel ion concentrations. 16959

3.8.4, 6.4.2

Rate of Dissolving of an Oxide Film on Aluminum. (In Russian.) N. P. Fedot'ev and I. Kosha-Shomodi. J. Applied Chem., USSR (Zhur. Priklad Khim.), **31,** No. 3, 497-500 (1958) March; Chem. Absts., **52,** No. 15, 12712 (1958) Aug. 10.

The rate of dissolving, ν mg/min/sq dm, of aluminum oxide film on aluminum in the process of its formation by oxidation in 10% sulfuric acid was studied. The film was removed from aluminum plates by a solution containing 20 g chromium trioxide and 35 ml orthophosphoric acid. The difference in weight of the aluminum plate, before oxidation and after the film was removed, gave the weight of the film. From the quantity of electric current required to form this amount of aluminum oxide, the theoretical amount of aluminum oxide was calculated. Empirically, the relation between ν and the temperature of the solution t was given by $\log \nu = 0.06t - 3.723$. The ratio of the actual surface (including that of the pores) and the apparent surface was directly proportional to the duration of oxidation. At 30 C, ν as a function of the sulfuric acid concentration passed through a maximum at 20% sulfuric acid.—ALL. 16947

3.8.4, 6.2.2

Distribution and Diffusion of Hydrogen in Iron. (In French.) Jacques Plusquellec, Pierre Azou and Paul Bastien. Compt. rend., 246, 3628-3631 (1958) June 30.

The distribution and diffusion of hydrogen in iron during electrolysis was studied. It was established that the hydrogen is located in the plane of the (112) family, a position which agrees with previous experimental data. If the electrolysis is stopped for a period of time, a strong absorption of hydrogen occurs when the electrolysis is resumed. —NSA. 16954

3.8.4, 6.3.5, 3.7.2

Effects of Alloying on the Kinetics of Oxidation of Niobium. W. D. Klopp, C. T. Sims and R. I. Jaffee. Battelle Memorial Inst. U. S. Atomic Energy Commission Pubn, A/CONF.15/P/712, 39 pp. Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958. Available from Office of Technical Services, Washington, D. C.

Kinetics of the air oxidation of pure niobium and of niobium binary alloys containing up to 25 at.% vanadium, 10 at.% molybdenum, 24 at.% chromium, 10 at.% tungsten, 35 at.% titanium and 45 at.% zirconium have been studied at elevated temperatures. Results of these studies have suggested several principles for the oxidation of niobium: a) ionic diffusion is rate-controlling under certain conditions when apparent linear oxidation is observed and b) alloying elements can effect the oxidation behavior by expanding or contracting the partially protective niobium pentoxide scale, by valence effects on diffusion rates through the scale and by formation of new scales. Pure niobium oxidizes linearly at 600 to 1200C. Moisture increased the oxidation rate at 600C. The scales consisted of an inner, adherent, oriented layer of niobium pentoxide containing traces of niobium monoxide and an outer, porous layer of niobium pentoxide. The oxide initially forms as adherent, protective niobium pentoxide but high compressive stresses developed as a result of the high oxide/metal volume ratio (2.69) cause the scale to crumble at a relatively low thickness. A steady state reaction ensues, with oxidation following the linear time law. On the basis of preliminary experiments it is believed that ionic diffusion, probably of oxygen ions, through the inner layer is the rate-controlling reaction. Alloying can affect oxidation behavior by three mechanisms: 1) Alloying additions with smaller crystal radii than that of niobium contract the niobium pentoxide promoting protective scales and parabolic oxidation, while larger ions expand the scale and increase the linear oxidation rates. 2) Valence of the alloying addition has a relatively minor effect on diffusion rates through the scale because of the large

number of defects in niobium pentoxide. 3) At alloying levels beyond the solubility limit of niobium pentoxide for the alloying ion, the oxidation rates are determined by the properties of new scales. (auth)—NSA. 16854

5. PREVENTIVE MEASURES

5.4 Non-Metallic Coatings and Paints

5.4.2, 6.4.2

Vitreous Enamelled Aluminum. (In German.) H. Kyri. Aluminum, **34,** No. 4, 213-216 (1958) April.

Information pertaining to the technology, uses and properties of vitreous enamelled aluminum is grouped according to the specific interests of the enamelling industry, the aluminum producers, fabricators and consumers. Accordingly, first, the chemical composition and characteristics of vitreous enamels, metal pretreatment, baking ovens, varieties and properties of the coatings, inspection, defects and economic aspects are discussed. Next, variations in the enamelling procedure according to the kind of aluminum material used, are examined. Aluminum of at least 99% purity requires but a rather simple surface treatment and permits the application of one coating only, without the possibility of touching up. Aluminum alloys of the aluminum-3% magnesium and aluminum-magnesium-silicon types require a chromate treatment and can be given several vitreous enamel coatings. Alloys containing more than 0.3% copper or more than 7% silicon are unsuitable for enamelling. The specific properties of this material make it particularly suitable for architectural purposes, interior decoration and marine uses. Similarly, it finds increasing acceptance in the manufacture of heating and cooking appliances, provided the enamel coated surface does not come into direct contact with food or beverages.—ALL. 16561

5.4.5, 4.6.11, 3.5.6

Corrosion in Sea Water of Steel Coated with Films of Petroleum Bitumens. E. I. Gurovich and A. S. Sosnina. Bulletin of the Academy of Science of the USSR, Technical Sciences Section (Izvest. Akad Nauk, Otdel. Tekh. Nauk,) **1957,** No. 4, 172-173; Chem. Abst., **51,** No. 21, 16262b (1957).

Various petroleum residues were tested as protective coatings of steel in sea water (Caspian Sea). The effectiveness of protection varied with the residues. The corrosion was lower than with uncoated steel plates; whatever corrosion occurred was traced to the film porosity, which permitted sea water to penetrate to the unshielded metal surface. The bituminous film swelled in sea water. The solvent used in applying the coating also affected the results, and the protection was much better from films formed by benzene solutions than from white spirit.—RPI. 16178

5.4.5

Single-package Wash Primers. E. S. Beck. Paint Ind. Mag., **73,** No. 5, 23-30 (1958).

The formation and properties of wash primers, both two-pack and, in more detail, single-pack, are discussed. The single-pack types are pigmented with zinc tetroxychromate/strontium chromate, lead chrome, chromium phosphate or barium chromate and their salt spray resistance properties are compared. In general, the two-pack standard type is superior, but the others have specific applications.—RPI. 16534

5.4.5

New Surface Coatings Based on Diisocyanates. M. E. Bailey. J. Soc. Plastics Engrs., **14,** No. 2, 41-43, 68-69 (1958) Feb.

Coatings are prepared by adding castor oil to diisocyanate in solvent under dry atmosphere and heating at 50-60 C; data for tolylene diisocyanate and diphenyl methane diisocyanate are given; suggested applications are protective coatings for dryer pans used in chemical plant, wooden plate and frame filter presses, steel racks for curing cement block, concrete floors; notes on earlier coatings developed in Germany are given.—BL. 16538

5.4.5, 2.2.5

The Role of Pigments in the Weathering of Automotive Paints. S. B. Twiss, W. L. Weeks and F. O. Thomas. Paper before Am. Soc. Testing Materials, Comm. D-1 Mtg., Atlantic City, June 18, 1957. Proc. ASTM, **57,** 1337-1366 (1957).

Correlation of certain properties of pigments with defects occurring during weathering of paints containing these pigments. Test panels of 28 different pigments were exposed for 1 year in southern Florida. Chalking consists of progressive shrinkage and erosion of paint film surface resulting in exposure and release of discrete pigment particles

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and small groups of particles. Effect creates surface whose roughness is related to pigment size, and whose morphology is determined by effective pigment size and shape. Bronzing is progressive shrinkage and degradation of paint surface which manifests itself in extensive release of sheets of material consisting of pigment and partially degraded vehicle. Microstructure of bulk surface is direct function of pigment size and shape. Checking is regarded as weathering defect due to degradation of vehicle to substantial depth of total paint film. Tables summarize chalking, bronzing and checking types of pigments, and pigments having combinations of these characteristics. Photomicrographs.—INCO. 16429

TECHNICAL REPORTS

on

PIPE LINE CORROSION

T-2 Statement on Minimum Requirements for Protection of Buried Pipe Lines. Prepared by a Special Task Group of NACE Technical Group Committee T-2 on Pipe Line Corrosion. Publication No. 56-15. Per Copy $.50.

TP-2 First Interim Report on Galvanic Anode Tests. (Pub. 50-2) NACE members, $3; Non-members, $5 Per Copy.

TP-3 First Interim Report on Ground Anode Tests. (Pub. 50-1) NACE members, $3; Non-members, $5 per copy.

T-2B Final Report on Four Annual Anode Inspections. A Report of Technical Unit Committee T-2B on Anodes for Impressed Current. Publication 56-1. Per Copy $1.00.

T-2B Use of High Silicon Cast Iron for Anodes. First Interim Report of Unit Committee T-2B on Anodes for Impressed Current. Publication No. 57-4. Per Copy $.50.

T-2C Some Observations of Cathodic Protection Potential Criteria in Localized Pitting. A Report of T-2C on Minimum Current Requirements for Cathodic Protection. Pub. 54-2. Per Copy $.50.

T-2C Some Obseravtions on Cathodic Protection Criteria. A contribution to the work of NACE Technical Unit committee T-2C. Publication No. 57-15. Per Copy $.50.

T-2C Criteria for Adequate Cathodic Protection of Coated, Buried, Submerged Steel Pipelines and Similar Steel Structures. A Report of NACE Technical Unit Committee T-2C on Criteria for Cathodic Protection. Pub. 58-15. Per Copy $.50.

T-2D Methods for Measuring Leakage Conductance of Coating on Buried or Submerged Pipe Lines—A Report of Tech. Unit Committee T-2D on Standardization of Procedures for Measuring Pipe Coating Leakage Conductance. Pub. 57-27, Per Copy $.50.

T-2G Tentative Recommended Specifications and Practices for Coal Tar Coatings for Underground Use. A Report of Technical Unit Committee T-2G on Coal Tar Coatings for Underground Use. Per Copy $.50.

T-2H First Interim Report. Tentative Recommended Specifications for Asphalt Type Protective Coatings for Underground Pipe Lines—Wrapped Systems. A Report of NACE Technical Unit Committee T-2H on Asphalt-type Pipe Coatings. Publication No. 57-11. Per Copy $.50.

T-2H Second Interim Report. Tentative Recommended Specifications for Asphalt-type Protective Coatings for Underground Pipe Lines—Mastic Systems. A Report of NACE Technical Unit Committee T-2H on Asphalt-type Pipe Coatings. Publication No. 57-14. Per Copy $.50.

T-2H Tentative Recommended Specifications for Asphalt-Type Protective Coatings for Underground Pipelines (Minimum Recommended Protection). A Report of NACE Technical Unit Committee T-2H on Asphalt Type Pipe Coatings. Pub. 58-12. Per Copy $.50.

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5.4.5

Epoxide Resins in Anti-Corrosion Finishes. F. L. Farr. Industrial Finishing, **9,** No. 114, 986-991 (1957).

The advantages of using epoxy resins in protective coatings for metal surfaces exposed to severe corrosive influences are discussed. Specific examples are given of the successful application of these materials to tank linings, ships hulls, structural steelwork, etc.—RPI. 16230

5.4.5, 1.3, 1.2.2

Lacquer Fights Corrosion with Success. I. Latzen. Chem. Rundschau, **11,** No. 5, 114-118 (1958).

An economic survey of protection against corrosion by means of paints is followed by a comprehensive review covering the protective paints used in various countries, the circumstances in which paints can be used against corrosion and requirements for their performance in the chemical, food, leather, textile and power industries, examples of special requirements and applications in industry, primers used for protective paints, coatings and used for higher temperatures and pretreatment of surfaces.—RPI. 16699

5.4.5

Organic Finishes Over Metal Surfaces. N. Perez. Plating, **45,** No. 3, 239-244 (1958).

A review of the development of organic coatings; drying processes; factors affecting adhesion and flexibility; epoxy resins; short notes on industrial applications of alkyd, urea, melamine, phenolic, vinyl, acrylic and epoxy resins. —RPI. 16702

5.4.5

Edge Protection. K. A. Van Oeteren. Deutsche Farben-Z., **12,** No. 2, 49-59 (1958).

The difficulty of adequately coating sharp edges of metal structures when painting is discussed, with reference to the effect of varying the viscosity, etc., of the paint. It is recommended that a special paint, of high pigmentation, be used for the edges alone.—RPI. 16736

5.4.5, 8.8.1

New Methods in Acid Plant and Corrosion Protection. (In German.) G. Schucking. Werkstoffe u. Korrosion, **9,** No. 5, 301-304 (1958).

The anti-corrosive properties are discussed, and the relative efficiencies tabulated, of a number of coating materials, inter alia polyisobutylene and other resin emulsions, with specific reference to use in acid plant.—RPI. 16670

5.8 Inhibitors and Passivators

5.8.4

Vapor-Phase Inhibitors. (In French.) Giorgio Mantovani. Corrosion et Anticorrosion, **6,** Nos. 7-8, 258-264 (1958) July-August.

A study of mopholine, camphor aniline and other inhibitors to protect metallic surface against atmospheric corrosion, especially before use of the metallic products. Water solubility, fusion point, boiling point and vapor tension.—BTR. 16548

5.8.4

Studies on Amine-Type Corrosion Inhibitors (12th Rept.) Adsorption Ability of Some Functional Groups. Pt. I. S. Fuji and K. Aramaki. Corrosion Engineering (Japan), **7,** No. 3, 37-41 (1958) May.

Study of a relationship between the adsorption of polar organic compounds on metal and their ability to inhibit corrosion in sea water. The corrosion inhibitive properties of compounds with straight hydrocarbon chains of 16 carbon atoms, such as acid amide, acid, alcohol, and amine were tested; amide ranked best, amine ranked second. Amide group shows increased adsorption as they have equal entent of covering effects of their alkyl groups. Our inhibitors (amine-acid complexes) show resembling behaviors with the amide.

High molecular weight sulfur-containing organic compounds seem to be effective in inhibiting corrosion of copper in sea water.—JSPS. 16483

5.8.4

Carbonate of Cyclohexylamine: Its Use as a Volatile Corrosion Inhibitor. (In French.) Products Chimiques, **60,** 471-473 (1957) Nov. 30.

Properties, including nontoxicity; applications to ferrous metals, either bare or plated with chromium, nickel, Monel metal, lead, zinc or aluminum; corrosion protection afforded machinery during marine transport, industrial equipment during extended shutdowns, machinery and parts subjected to wear.—MR. 16246

6. MATERIALS OF CONSTRUCTION

6.6 Non-Metallic Materials

6.6.6

The Reaction Between Glass and Aggressive Media. A. F. Zak. J. Applied Chem., USSR (Zhurnal Prikladnoi Khimii), **30,** No. 9, 1292-1298 (1957). Available from Associated Technical Services, Inc., P. O. Box 271, East Orange, New Jersey.

Chemical stability of glass fibers is found to depend on the composition of the glass, replacement of silicon dioxide by small amounts of acid or alkaline earth oxides often increasing stability to acids and water. 6 figures, 8 references. —ATS. 16350

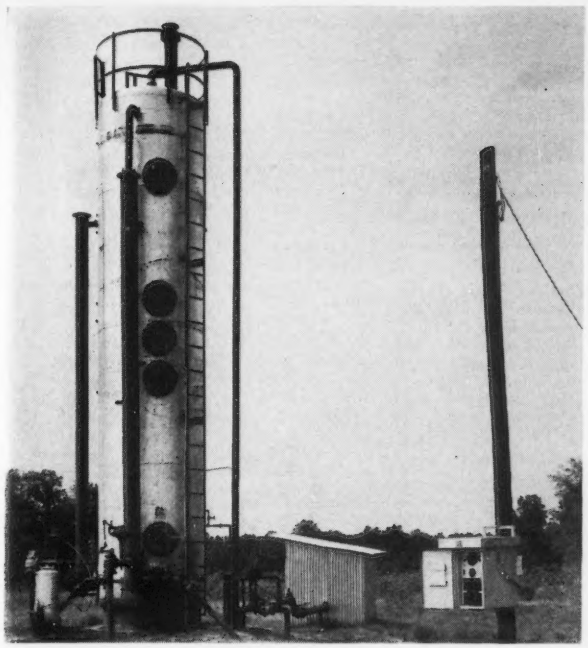
6.6.8, 8.8.3

Polyvinyl Chloride Pipe Handles Corrosive Plating-Solutions. T. F. Long. Plating, **44,** No. 9, 975-978 (1957).

The properties of polyvinyl chloride and the use, application, and advantages of polyvinyl chloride pipe in the U. S. electroplating industry are surveyed.—MA. 16210

6.6.11, 3.3.3

Relationship Between Limnoria Species and Service Life of Creosoted Piling. H. Vind, H. Hochman, J. Maraoka and J. Casey. Paper before Am. Soc. for Testing Materials, 2nd Pacific Area Nat. Mtg., Symp. on Wood for Marine Use and Its Protection from Marine Organisms, Los Angeles, Sept. 17, 1956. ASTM Special Technical Pubn. No. 200, 1957, 35-49; disc. 49-50.



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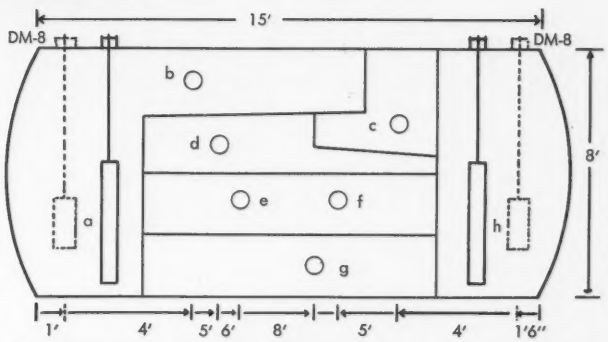

This drawing depicts anode placement for both magnesium and graphite.

ANODE	AREA	CURRENT
a	175 sq. ft.	1.75 Amps
b	100 sq. ft.	1.00 Amps
c	78 sq. ft.	.78 Amps
d	194 sq. ft.	1.94 Amps
e	97 sq. ft.	.97 Amps
f	97 sq. ft.	.97 Amps
g	154 sq. ft.	1.54 Amps
h	165 sq. ft.	1.65 Amps
	Total	**10.60 Amps**

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As protection against marine borers, creosote is less effective in tropical than in temperate or cold water harbors. Differences between species of marine borers prevalent were considered as a possible explanation for differences in serviceability of treated timbers in respective areas. Creosoted piles that were removed from Heuneme Harbor (Calif.) after 13 years of service were studied. Findings showed that species Limnoria tripunctata initiates early failure of creosoted piling in California harbors and other southern ports. Leaching by sea water removed most toxic ingredients of creosote from outer layer of piling. These more toxic ingredients were diffused both inward and outward by action of sea water. Apparently ingredients of creosote which were not readily leached by sea water were less toxic to Limnoria tripunctate than to other species of marine borers. Photos.—INCO. 16407

TECHNICAL REPORTS

on

REFINING INDUSTRY CORROSION

T-8 High-Temperature Hydrogen Sulfide Corrosion of Stainless Steel—A Contribution to the Work of NACE Tech. Group Committee T-8 on Refinery Industry Corrosion, by E. B. Backensto, R. E. Drew, J. E. Prior and J. W. Sjoberg. Pub. 58-3, Per Copy $.50.

T-8 Compilation and Correlation of High Temperature Catalytic Reformer Corrosion Data—A Contribution to the Work of NACE Tech. Group Committee T-8, by G. Sorell. Pub. 58-2, Per Copy $.50.

T-5B-2 Effect of Sulfide Scales on Catalytic Reforming and Cracking Units. Part 1—Metallographic Examination of Samples From a Catalytic Reforming Unit. Part 2—Intergranular Corrosion of 18-8 Cr-Ni Steel as a Result of Hydrolysis of Iron Sulfide Scale. A Contribution to the Work of NACE Task Group T-5B-2 on Sulfide Corrosion at High Pressures and Temperatures in the Petroleum Industry. Per Copy $.50.

T-5B-2 Collection and Correlation of High Temperature Hydrogen Sulfide Corrosion Data—A Contribution to the Work of NACE Task Group T-5B-2 on Sulfide Corrosion at High Temperatures and Pressures in the Petroleum Industry: From M. W. Kellogg Co., New York by G. Sorell and W. B. Hoyt. Pub. 56-7. Per Copy $1.50.

T-5B-2 High Temperature Hydrogen Sulfide Corrosion in Thermofor Catalytic Reformers—A Contribution to the Work of NACE Task Group T-5B-2 on Sulfide Corrosion at High Temperatures and Pressures in the Petroleum Industry: From Socony Mobil Oil Co., Inc., Res. & Dev. Lab., Paulsboro, N. J. by E. B. Backensto, R. D. Drew, R. W. Manuel and J. W. Sjoberg. Pub. 56-8. Per Copy $1.00.

T-5B-2 Effect of Hot Hydrogen Sulfide Environments on Various Metals—A Contribution to the Work of NACE Task Group T-5B-2 on Sulfide Corrosion at High Temperatures and Pressures in the Petroleum Industry: From Sinclair Res. Lab. Inc. Harvey, Ill. Pub. 57-2. Per Copy $.50.

T-5B-2 High Temperature Sulfide Corrosion in Catalytic Reforming of Light Naphthas—A Contribution to the Work of NACE Task Group T-58-2 on Sulfide Corrosion at High Temperatures and Pressures in the Petroleum Industry: From Humble Oil & Ref. Co., Baytown, Texas. Pub. 57-3. Per Copy $.50.

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6.7 Duplex Materials

6.7.2

Alumina-Base Cermets. Pt. 5. J. G. Stradley and T. S. Shevlin. Ohio State Univ. Res. Foundation for U. S. Wright Air Development Center, U. S. Air Force. Feb., 1958, 15 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 151143).

A non-combustible helium-hydrogen atmosphere produced compact, nonporous cermet specimens with fired properties reported to be equal or better than the metal alone. For the tests, solid solutions of aluminum oxide-chromic oxide and aluminum oxide-nickel monoxide spinels were combined with iron metal and their physical properties studied. The helium-hydrogen atmosphere was selected with the belief that a pure hydrogen atmosphere produced too much reduction of the reducible portion of the oxide phase and helium or vacuum produced too little. It was felt that for sufficient bonding to occur to produce dense specimens, some reduction of the secondary oxide to metal must take place. The metal, in turn, would alloy with the original metal and provide a better bond between oxide and metal phases. The secondary oxides chromic oxide and nickel monoxide were reduced and alloyed with the original metal. Specimens were obtained with modulus of elasticity values equal or better than the accepted value for iron alone.—OTS. 16667

6.7.2, 3.8.4

Predicting the Thermodynamic Stabilities and Oxidation Resistances of Silicide Cermets. Alan W. Searcy. Uni. California. J. Am. Ceram. Soc., **40,** 431-435 (1957) Dec.

Available information on the thermodynamic stabilities of transition metal silicides is summarized and is used to demonstrate the calculation of the stability of silicide-metal mixtures. The cause of the outstanding oxidation resistance of certain silicides is analyzed and the results are used to predict high oxidation resistance for several untested disilicides. Thermodynamic and structural arguments are applied to the prediction of compositions of thermodynamically stable and oxidation-resistant cermets. (auth)—NSA. 16333

7. EQUIPMENT

7.4 Heat Exchangers

7.4.1, 8.4.5, 1.3

Heat Transfer Problems in Reactor Technology. (In German.) W. Junkermann. Nukleonik, **1,** 73-87 (1958).

A survey is presented on heat transfer problems in reactors. The requirements for reactor coolants and possible structural materials for heat exchangers are indicated. The heat equations for various coolants are derived. Special properties of coolants such as air, water and gases are described. Heat exchangers are classified and their construction is described. Technological and corrosion problems for various reactor types are discussed, 28 references.—NSA. 16570

7.6.4, 3.2.2, 3.8.4, 3.5.7

Recent Research on the Corrosion of Boiler Tubes. S. G. Clarke. Paper before Soc. Chemical Industry, Manchester, March 28, 1958. Chem. & Ind., No. 22, 647-648 (1958) May 31.

Description of build-up of dense local deposits of magnetic iron oxide on internal surfaces of the steam-generator tubes of boilers working above 350 psi. At higher pressures, especially above 1000 psi, pitting is associated with permanent embrittlement of adjacent steel, and in extreme cases a "window" of metal is ejected from the tube and a prolonged explosive generation of steam is the result. A new mechanism was proposed for formation of magnetic iron oxide, whereby action propagates at oxide/metal interface by diffusion of hydroxyl ions inwards through film as it forms. This suggests that corrosion might be prevented by controlling defect structure of magnetite by attention to certain minor constituents of the steel. An example was given where N_2 ions in magnetite lattice would be detrimental. A corrosion-testing apparatus was described, designed to reproduce corrosion under controlled conditions of water and metal composition, pressure and heat-transfer rate.—INCO. 16443

7.6 Unit Process Equipment

7.6.4

Spherical-Head Feedwater Heater. A. E. Pickford and J. M. Phelps. C. F. Braun and Co. Power, **102,** No. 3, 103, 109 (1958) March.

New spherical-waterbox all-welded heater is designed to avoid leakage and high maintenance and can be installed horizontally or vertically. The 3000-psi feedwater heater makes 2 passes through U-tube bundle. Tube sheet is an integral part of sphere. Tubes are 5/8-in., 18-gauge Monel; tube bundles are about 40 in. diameter and 30 ft long. Stainless steel impingement surfaces opposite nozzles in distributor avoid eliminating tubes to make room for bolted impingement plate. Diagram.—INCO. 16707

7.6.6

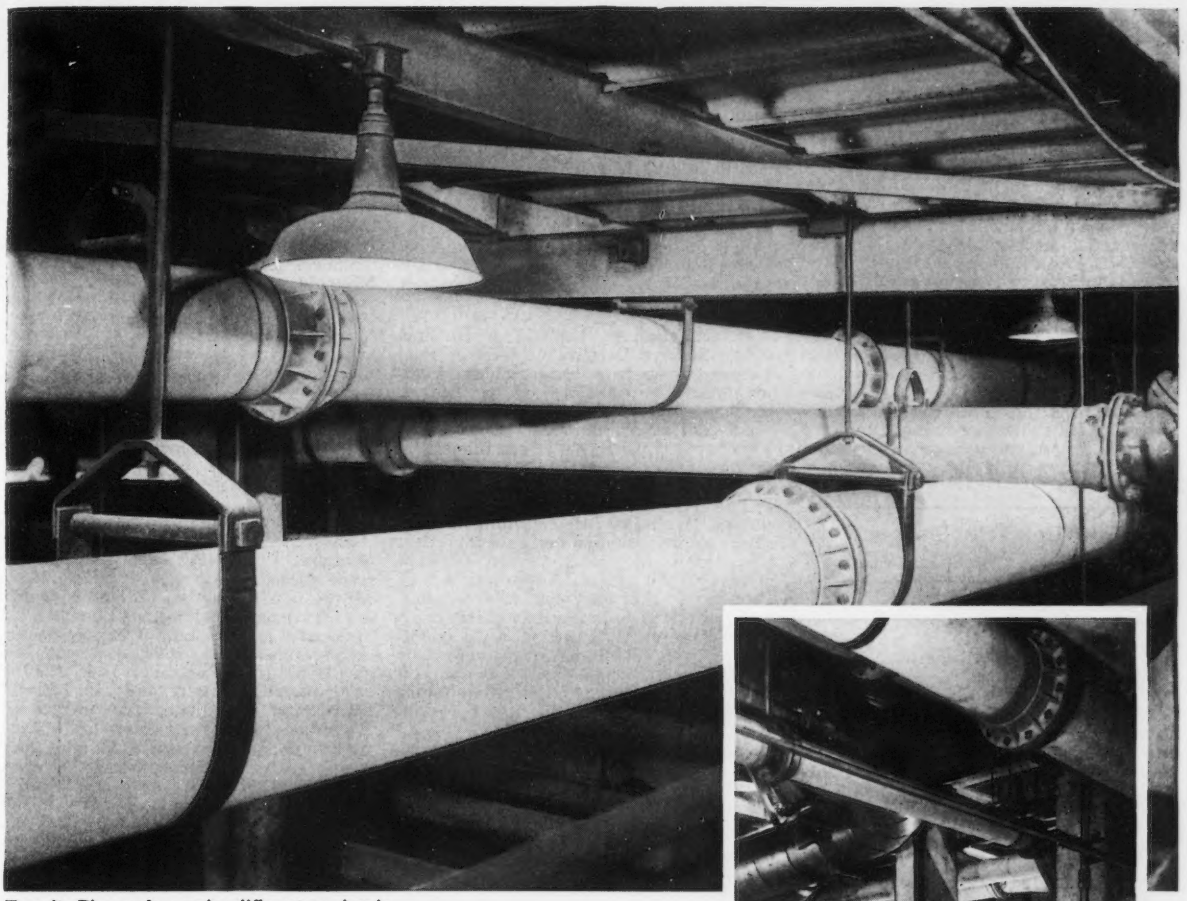
Second Report—Regional Digester Activity. C. W. Smith. Paper before TAPPI, 11th Eng. Conf., Boston, October 8-11, 1956. TAPPI, **40,** No. 9, 728-729 (1957) Sept.

TAPPI mill report forms from 21 mills covering 162 digesters show corrosion rates considerably lower than those in First Report—No. 9, 1955. Average corrosion rate on 152 C-steel digesters ranged 0-110 mpy with median of 28.0 mpy. For 4 Inconel-clad digesters rates ranged 0-40.0 mpy with median of 11.5 mpy; and for a Type 316-clad digester, average corrosion rate was 20.0 mpy. Tables.—INCO. 16294

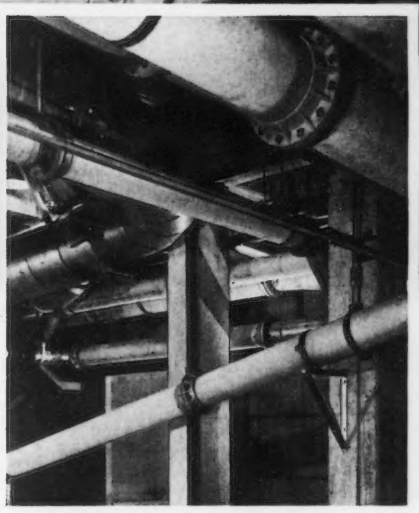
7.6.6, 5.4.3

Modern Sulphite Digester Linings—Recent Technical Developments. E. F. Tucker. Paper before Superintendents Assoc., 38th Annual Convention, Chicago, May 21-22, 1957. Paper Trade J., **141,** No. 42, 25-30 (1957) October 21.

History of development of digester and its linings. There are in operation in North America at present about 600



Transite Pipe performs nine different services in the St. Regis Company's Jacksonville Plant.



Transite line is from new mill washed stock chest to old mill washed stock chest.

Transite Pipe helps St. Regis work giant new "Seminole Chief" at full capacity!

Chief Engineer, L. C. Crowder, tells of experience with Transite . . . how its flow capacity and "cleanliness" determined its selection

Almost by the day, we see new reports on the added economy and performance that Transite® Pipe brings to water and stock lines. One such report comes to us from the St. Regis Company's Jacksonville Plant:

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141a

digesters of type discussed of which 13 have some type of acid-resisting chromium-nickel steel lining or cladding fabrication. Problem of stress in steel shell is discussed. Extensometer for strain gauge measurements is described. Strain gauge stations are placed for measuring both vertical and horizontal strains by welding Type 316 stainless buttons to shell of digester. Brick lining in a digester is an economically sound item of equipment which has been a basic and indispensable factor in building of a great industry. Alloy-lined or clad digesters represent an ideal, equipment-wise, on which much time, money and hard work must be expended before perfection is reached. Meanwhile, we must depend on brick linings now and for some time to come; and an intelligent understanding of just what we have and must use in the form of a steel shell and its protective lining can make sulfite mill operation much easier and results more profitable. Diagrams, graphs.—INCO. 16371

7.7 Electrical, Telephone and Radio

7.7, 5.3.4

High-Power Cables with Extruded Aluminium Sheaths. H. J. Hilgendorff and W. Thele. F. G. Rundschau, **42,** 50-62 (1957).

The development of the corrugated aluminum sheath is described, its structure and productions outlined. The lays, make-up and dimensions of cables are given with coper equivalents, weights and costs. Protectors against corrosion, carriage and laying are also considered. —MA. 15852

7.7, 3.5.9

Magnetic Materials in Ultrahigh Temperature Electronic Transformers. H. B. Harms. Paper before Am. Inst. Elec. Engrs., Winter General Mtg., New York, Feb. 2-7, 1958. Electrical Eng., **77,** No. 5, 408-412 (1958) May.

U.S. Air Force is sponsoring development of electronic power transformers that will be capable of withstanding 500 C ambient temperature as well as other adverse environments. Description is given of experimental work to determine quantitatively the effect of temperature on magnetic core materials. Core loss, excitation and incremental permeability were measured on a grain-oriented silicon iron at several flux densities and at 4 different temperatures up to 550 C. Steel evaluated was AISI-M6 grain-oriented 3¼ silicon steel. Graphs.—INCO. 16515

8. INDUSTRIES

8.4 Group 4

8.4.5

Full-Scale OMR Plant. E. F. Weisner, p. 197-209 (of) Proceedings of the SRE-OMRE Forum Held at Los Angeles, California, February 12 and 13, 1958. Atomic International Div., North American Aviation, Inc., Canoga Park, Calif. U. S. Atomic Energy Commission Pubn., TID-7553 (p. 197-209), 13 pp.

The concept of the Organic Moderated Reactor is set forth and a description is given of a 12.5 megawatt electrical power plant employing such a reactor. This particular plant has steam conditions of 450 psia and 550 F with a flow of 162,000 lb/hr. The outlet organic temperature is 625 F. The fuel consists of 6200 kg of aluminum-clad uranium alloy enriched to 1.8% U^{235}. The research and development program is discussed. Full-scale tests of various fuel element types, control-safety rods and fuel handling techniques are in progress. Process systems such as residue handling, degasification, pressurization, purification and steam generation are being studied. Corrosion tests and studies of physical properties of organics under varying conditions are being conducted on a continuing basis. (auth.)—NSA. 16701

8.4.5

Investigation of the Effect of Fuel Filler Surface Conditioning Upon Fuel Element Properties. Stuart Weiss. Westinghouse Electric Corp. U. S. Atomic Energy Commission Pubn., WAPD-NCE-2261, Nov. 9, 1956 (Declassified May 2, 1957), 8 pp. Available from Office of Technical Services, Washington, D. C.

The results of two test runs conducted with surface-conditioned fuel fillers are reported to evaluate the effect upon diffusion zone thickness, bond strength and corrosion properties of Zircaloy-2 clad uranium-10wt.% niobium. Results indicate that there is no significant effect of fuel surface condition upon diffusion zone thickness. The bonding interface of the rolled fuel element has greater strength than either the clad or the rod. The corrosion data were inconclusive because of the small number of tests, the uncontrolled variables and the erratic corrosion properties of the particular uranium-niobium alloy used.—NSA. 16427

8.4.5

Test Program for PAR In-Pile Slurry Loop Equipment. Lester J. King. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-58-6-11, June 4, 1958, 9 pp. Available from Office of Technical Services, Washington, D. C.

The major portions of the test program required for demonstrating the operability and dependability of the PAR in-pile slurry loop complex are listed. The responsibility for making the tests is divided to give Westinghouse the responsibility for assembling the loop, loop auxiliaries, and controls and demonstrating their satisfactory operation out of pile and to give ORNL the responsibility for testing radiation monitoring systems and instruments, carriers and tools for handling the radioactive loop and samples, and loop sedmenting equipment. The program for test runs at the reactor site is outlined. (auth)—NSA. 16549

8.4.5

Dissolution and Feed Preparation for Aqueous Radiochemical Separation Processes. F. L. Culler and R. E. Blanco. Oak Ridge Natl. Lab. U.S. Atomic Energy Comm. Pubn., A/CONF.15/P/1930, 85 pp. Prepared for the 2nd U.N. International Conf. on the Peaceful Uses of Atomic Energy, 1958. Available from Office of Technical Services, Washington 25, D. C.

Most irradiated power reactor fuels can be radiochemically processed by the well established solvent extraction methods provided that the corrosion resistant cladding materials can be removed or dissolved and that the fuel element cores can be dissolved in or converted to an aqueous solution highly salted with nitrate anions. Thus, the aim of much of the current aqueous processing development is to devise compatible mechanical and chemical dissolution procedures for all power reactor fuels. Mechanical processing is being studied as an alternate to non-nitric-acid head-end systems to facilitate associated waste storage and corrosion problems. Mechanical operations include disassembly to complex fuel bundles and dejacketing of fuel rods and pins to expose nitric acid soluble cores. Mechanical techniques under study include chopping, roll decladding, sawing and breaking. Chemical removal of zirconium cladding can be done with aqueous ammonium fluoride, hydrofluoric acid, sulfuric acid, or by anhydrous hydrochlorination or chlorination in gas-solid or anhydrous organic media. Stainless steel may be declad by dissolution in sulfuric acid. (auth)—NSA. 16789

8.4.5

Light Water Moderated Reactors in Cross Flow. Kidde (Walter) Nuclear Labs., Inc. U. S. Atomic Energy Commission Pubn., NYO-3924, Sept. 30, 1953 (Declassified February 27, 1957), 90 pp. Available from Office of Technical Services, Washington, D. C.

Results are reported from exponential measurements at BNL on water-reflected lattices of uranium rods 0.750 in. in diameter, 4 ft. long, and jacketed with 30 mils of aluminum. Experiments are described for determination of the average heat-transfer coefficient, the local heat-transfer coefficients, and the on-set of nucleate boiling for water flowing across a bank of staggered rods at high Reynolds numbers; the effect of thermal cycling on rod-to-jacket transfer for an unbonded jacket was also investigated. Results are presented from an investigation of the hydrodynamic effect of high-velocity water flowing perpendicularly to a bank of rods arranged in a triangular lattice. The preparation of long zirconium-clad uranium fuel rods and the corrosion testing of samples of annealed zirconium tubing in distilled water and solutions of H_3BO_3 and $K_2B_4O_7$ are discussed.—NSA. 16217

8.4.5

Control Problems in Sodium Cooled Graphite Moderated Reactors. J. E. Owens. Paper before 2nd Nuclear Eng. and Science Conference, Philadelphia, March 11-14, 1957. Am. Soc. Mechanical Engrs., Paper No. 57-NESC-77, 1957, 11 pp.

Primary purpose of Sodium Reactor constructed by Atomics International is to obtain all possible information about performance of reactor core and its auxiliary equipment. Uranium fuel element is clad with stainless steel and reactor structure is of stainless steel. Boron-nickel rods are used in Safety and Shim of control system of reactor. Diagrams, photos, graphs, tables.—INCO. 16254

8.4.5

The Homogeneous Reactor Experiment No. 2. S. E. Beall and W. D. Burch. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., A/CONF 15/P/449, 50 pp. Available from Office of Technical Services, Washington 25, D. C.

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

The Homogeneous Reactor Test is a 5 Mw aqueous homogeneous reactor. The purpose and design details were

presented in Paper 526A of the 1955 Geneva Conference. The present paper relates the experiences with this reactor through the construction, engineering testing and startup phases. Construction and hydrostatic testing of the reactor container were completed in the fall of 1955 and the reactor and chemical plant components were installed from October 1955, to May 1956, at which time engineering testing of the systems began. These tests were interrupted to replace stainless-steel flanges and leak-detector tubing that were damaged by stress-corrosion cracking resulting from chloride contamination in the leak-detector system. The engineering tests were completed in December 1957, and reactor operation was begun in January 1958. The engineering tests, critical experiments and operation of the reactor at power are described. Similar information is also presented for at various temperatures between 200-350 C. Corrosion equations have been developed at HAPO to express the corrosion rate of aluminum in and out-of-reactor. The equations can be modified to express the corrosion rate of aluminum in either inhibited or moderately corrosive media such as Hanford process water. From these equations operating curves may be drawn which may be used to determine limits for the case of aluminum in water cooled reactors. (auth)—NSA. 16754

8.4.5, 2.3.9

Ultrasonically Accelerated Dissolution of Corrosion-Resistant Metals. J. L. Straughn, M. L. Bromberg and W. B. Tarpley. Aeroprojects, Inc. U. S. Atomic Energy Commission Pubn., NYO-7920, March, 1958, 83 pp. Available from Office of Technical Services, Washington, D. C.

Ultrasonic energy was applied to the dissolution of candidate heterogeneous reactor fuel-element cladding metals presently difficult to dissolve, with the objective of increased dissolution rate and decreased concentration of corrosive dissolvent components. Significant increases in dissolution rates were achieved with austenitic stainless steels, Zircaloy-2 and molybdenum, and dissolution was promoted in reagents that do not normally attack these metals. Three basic approaches to ultrasonically accelerated dissolution were investigated: bulk activation of a solution, activation of a dissolving surface in close proximity to the ultrasonic coupler, and direct activation in which the metal itself is flexurally vibrated in a dissolving solution. Studies with pressurized systems demonstrated the important role of cavitation in both bulk-activation and close-proximity treatment, since dissolution rate was significantly reduced as cavitation was suppressed. The close-proximity approach was the more effective, and Zircaloy-2 dissolved in 4 **M** nitric acid-13.5 **M** sulfuric acid solution by this technique showed a fourfold rate increase over non-ultrasonic dissolution. Greatest improvement in dissolution was achieved by direct ultrasonic activation of the metal. In a mixture of 4 **M** nitric acid-0.1 **M** hydrochloric acid dissolution of Type 304 stainless steel was increased approximately 200-fold to an acceptable rate of 4 mg/cm²/min, whereas a non-ultrasonic control showed negligible dissolution in this reagent. The possibilities of reduced equipment corrosion and concentration of corrosion attack as desired have important implications. Prototype all-metal dissolvers for successful containment of dissolvents were constructed, the vibratory energy being introduced into corrosive environments without mechanical motion and its concomitant sealing difficulties. (auth)—NSA. 16734

8.4.5, 3.5.4

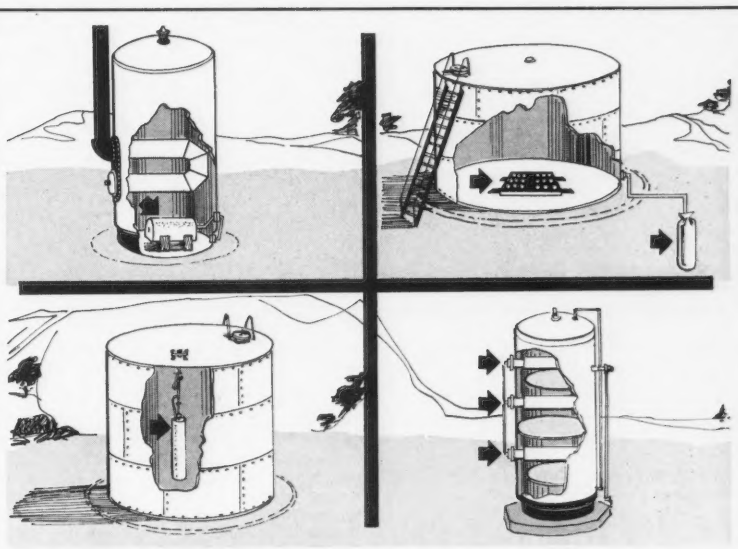
Review of Loop Programme in the NRX Reactor. (Paper presented at) Sixth UK/Canada Technical Conference, Chalk River, Ontario, October 21-22, 1957. R. F. S. Robertson, September, 1957, 24 pp. Available from the Scientific Document Distribution Office, Atomic Energy of Canada, Ltd., Chalk River, Ontario, Canada.

The over-all loop program is reviewed briefly. Results obtained from investigations of the corrosion of mild and stainless steels in these loops and some conclusions reached regarding the activation and distribution of activated corrosion products around a loop system are discussed. The escape and distribution in the loop circuit of fission products from defected uranium dioxide fuel rods under irradiation are discussed. (auth)—NSA. 16300

8.4.5, 3.5.4

Effects of Neutron Irradiation on Metals and Alloys. A. H. Cottrell. Atomic Energy Research Establishment, Harwell Berks, Eng. J. Brit. Nuclear Energy Conf., **3**, 50-67 (1958) Jan.

The behavior of metals and alloys under neutron irradiation in a reactor is investigated. An explanation of how damage originates from the collision and absorption of neutrons by nuclei, and a description of the behavior of the fast ions, produced from these neutron encounters, as they penetrate the sur-



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rounding material are given. The estimated amount of damage is compared with measured values. Annealing of the damage in which displaced atoms gradually migrate back into lattice positions under the action of thermal agitation is discussed. The effects of irridiation on the mechanical properties of metals and alloys are discussed. Radiation growth in uranium and the effects of irradiation on the corrosion resistance of metals are discussed.—NSA. 16445

8.4.5, 3.5.4

Summary of Bettis Irradiation Data on Reactor Fuel Structural and Control Rod Materials. R. H. Fillnow. Westinghouse Electric Corp. U. S. Atomic Energy Commission Pubn., WAPD-A1W(M)-366 (Del. 2), July 1956 (Declassified with Deletions March 18, 1957), 35 pp. Available from Office of Technical Services, Washington, D. C.

The objective of irradiation studies performed at the Bettis Atomic Power Division has been to evaluate suitable nuclear fuels, control rod materials and structural materials for use in the Pressurized Water Reactor (PWR). In general, irradiation induced physical property changes were measured on subsize specimens, exposed in reactors such as the Materials Testing Reactor (MTR) for irradiation periods that were equal to or greater than the expected reactor design exposure. Irradiation studies were made on both clad and unclad samples of fuel materials. Irradiation tests of experimental assemblies of the above materials were performed, whenever possible, under conditions that duplicated the corrosive environment and temperatures inherent to the pressurized water reactor design. The discussion of irradiation test results is divided into four separate sections: uranium base alloys, uranium dioxide, structural materials and control rod materials. (auth)—NSA. 16181

8.4.5, 4.7

Fluid Metal Fuels: UO_2-NaK Slurry. B. M. Abraham, H. E. Flotow and R. D. Carlson. Argonne National Lab. U. S. Atomic Energy Commission Pubn., A/CONF.15/P/457, 14 pp. Available from Office of Technical Services, Washington 25, D. C.

Prepared for the Second U.N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Investigations of UO_2-NaK suspensions were initiated as part of a program to develop a fluid suitable for a fast reactor. Most of this work was done with suspensions containing 5 volume % (40 wt. %) uranium dioxide in sodium-potassium, a concentration suitable only for thermal reactors, because the necessary basic information can be obtained more readily with dilute slurries. As normally prepared, uranium dioxide contains more than the stoichiometric amount of oxygen. It was found that sodium-potassium will readily wet UO_{2+x}, a necessary condition to effect suspension, if x is no greater than 0.01; if x is greater than 0.01 wetting occurs after the excess oxygen has reacted to produce sodium oxide. The minimum contact angle observed for sessile drops of sodium-potassium on uranium dioxide plaques was 22°. This result implies that the slurries will be flocculated. A slurry of 5 volume % uranium dioxide could be maintained uniform throughout the loop volume at a minimum flow rate of 2ft/sec (61 cm/sec) at all temperatures up to 500 C. Above 500 C., the slurry could no longer be maintained uniform at flow rates of 8 ft/sec (244 cm/sec). As soon as the temperature was reduced to 480 to 490 C the slurry became uniform in a matter of seconds. There was no marked change in the particle size of the uranium dioxide after circulating for 400 hours at the high temperatures. Metallographic examination of loop sections and chemical analyses of the slurry components failed to show evidence for corrosion or erosion. Some preliminary work with a 15 volume % slurry (65 wt. % uranium dioxide), which begins to approach the concentration required for a fast reactor, showed that the slurry was sufficiently fluid to be circulated in the apparatus and it appeared to be uniform. Information obtained to date (in the absence of radiation) on the rheology, the corrosion and the erosion of the slurries indicates that dilute suspensions of uranium dioxide in sodium-potassium offer promise as thermal reactor fuels; more information is needed on concentrated slurries before a definite statement can be made on applicability to fast reactors. (auth)—NSA. 16774

8.4.5, 6.2.5

Investigation of Materials for a Water Cooled and Moderated Reactor. Louis Scheib. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., ORNL-1915 (Del.), August 1954 (Declassified with Deletions May 8, 1957), 169 pp. Available from Office of Technical Services, Washington, D. C.

An investigation of the materials for use in the water-moderated and cooled Army Package Power Reactor (APPR) operating at about 500 F was made. The available literature was analyzed and the results of the different investigators were compared and averaged. Twenty different materials, including stainless steels, nickel alloys, Stellites and others, were investigated from the point of view of physical properties, susceptibility to radiation damage and corrosion resistance. Corrosion rates were established for all the materials under various conditions, such as irradiation, flow, weld, stress and various water conditions. Type-304 stainless steel was selected as the basic structural material. Operating conditions, to maintain minimum corrosion, were established also. (auth)—NSA. 16315

8.4.5, 6.4.2, 3.4.3, 3.8.4

The Use of Aluminum as Fuel Cladding in High Temperature Water Cooled Reactors. J. A. Ayres, R. L. Dillon and R. J. Lobsinger. General Electric Co., Hanford Atomic Products. U. S. Atomic Energy Commission Pubn., A/CONF. 15/P/1430, 9 pp. Available from Office of Technical Services, Washington, D.C.

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Aluminum corrosion product solubility has been found to be an important factor in aqueous aluminum corrosion. High aluminum corrosion rates in dynamic tests, as compared to static test, can be accounted for in terms of oxide solubility. Hanford solubility measurements have established the solubility of aluminum corrosion product in the temperature range 200 to 350 C. Corrosion rates in dynamic systems have been successfully predicted on the basis of corrosion product solubility, sample area and the rate of water refreshment. In the limiting case of very high water throughput and/or low sample area, however, the rate is not controlled by the oxide solubility. Instead the rate of corrosion depends on the rate of the dissolution process. Limiting (maximum) dissolution rates in deionized water have been determined in the chemical plant, which is attached to the reactor system, although in a separate shielded cell, to demonstrate the continuous purification of the fuel solution. The performance of the chemical plant is discussed with respect to the continuous removal of the insoluble corrosion and fission products. Also described is the manner in which the recovered solids are processed to remove the heavy water and to accomplish solution in a light water, sulfuric acid medium, so that a homogeneous sample of the removed material can be used for evaluation of the plant. The development of remote maintenance techniques, the maintenance experience and process improvements are described in detail. (auth)—NSA. 16753

8.4.5, 6.6.6

Use of Ceramics in Power Reactors. C. E. Curtis. Ceram. Ind., **71**, No. 1, 76-79 (1958) July.

Discussion of possibility of a reactor design with the fuel, moderator and breeder blanket in ceramic form and with gas as a coolant. Such a ceramic reactor could be operated at much higher temperatures than reactors consisting partly of liquids and it is at these high temperatures, above 1000 C, that ceramics are most useful while metals are apt to be unusable. Damage to ceramics caused by irradiation at lower temperatures is reported to be healed automatically at high temperatures; this is not true of most other materials. Corrosion problems in ceramics in contact with gases at high temperatures are, in general, much less acute than those encountered in the presence of liquids. Tables, diagrams.—INCO. 16791

8.4.5, 8.9.5, 1.2.5

Simulation of Various Accident Considerations for a Merchant-Ship Pressurized-Water Reactor. R. S. Boyd and others. Battelle Memorial Inst. for U. S. Atomic Energy Commission, August 1958, 70 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order BMI-1269).

This study was made in connection with development of the "Savannah" the first nuclear merchant ship. Analyses were made of the response of the nuclear merchant ship reactor to four foreseeable accident conditions: continuous rod withdrawal, startup, loss of coolant flow and cold-water insertion. Analog simulation techniques were used. Three phases of operation were considered in these analyses: operation with no safety system, high-flux scram and period scram. The programs involved the variations of the scram parameters and the reactor temperature coefficients over the range of possible values for the reactor power plant. Results, according to the report, indicated that the inherent stability of the reactor system is sufficient to attenuate the power excursions resulting from the first three accidents. In the cold-water accident, if the difference in loop temperatures is on the order of 300 F, scram was found necessary to limit the power excursion.—OTS. 16741

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8.8 Group 8

8.8.1, 6.6.8, 4.3.3, 5.4.5

Corrosion Problem at An Alkali-Chlorine Plant. J. F. Bosich. Diamond Alkali Co. Ind. and Eng. Chem., **50,** No. 7, 69A-70A (1958) July.

Liberal use of plastics helps solve difficult corrosion problems at Diamond Alkali Co. Plain steel chutes lined with polyvinylchloride convey sodium bicarbonate to prevent corrosion and product contamination and to keep material from adhering to metal. Stainless steel, formerly used, did not work as well. A modified epoxy coating is used to line a steel carbon dioxide separator in place of a stainless steel separator that failed. Underground piping and storage tanks are protected from corrosion by cathodic protection. Caustic liquor cooler tank fabricated of nickel clad steel is used to store 50% caustic soda at 180 F. Leakage of caustic occurred at weld seams of tank due to stress corrosion. Weldments of tank were coated with neoprene and tank has been in service over three years with no further leaks in evidence. Hoppers used to separate coarse from fine milk of lime at 175 F were fabricated from 403 stainless steel. Long hairline cracks developed on bottom of hopper after only six weeks operation. An attempt to weld the cracks was unsuccessful. A steel plate welded on bottom and lined with neoprene resulted in greatly increased efficiency of operation. Photos. —INCO. 16737

8.8.3, 5.3.4

Electroforming of Waveguide Components for the Millimeter-Wavelength Range. A. A. Feldmann. National Bureau of Standards. NBS Circular No. 587, Nov. 15, 1957, 13 pp. Available from U. S. Dept. of Commerce, Washington 25, D. C.

Simple techniques are outlined which permit production of high-quality millimeter components by electroforming with minimum of equipment, time, or specialized knowledge of electrodeposition. Silver, copper and nickel are used; silver and copper account for 95% of all millimeter waveguide components; nickel is used where components might be exposed to very high temperatures or chemical attack. Equipment required, mandrels, cleaning process, masking of mandrels, electrodeposition and post-forming operations are discussed. Nickel, silver and copper plating solutions are described. Nickel is deposited directly on zincated aluminum mandrel. If laminated structure is desired, nickel can be plated on top of 0.015-0.020 in. deposit of copper or silver. Causes and suggested cures are given for poor adhesion, rough deposits and treeing, dark spots and pitting. Pitting happens most frequently with nickel baths, hydrogen being considered most common cause. Table lists electrochemical equivalents and thickness factors.—INCO. 16207

8.8.5, 6.2.2

Progress in Iron Castings. H. Bornstein. 1957 Charles Edgar Hoyt Memorial Lecture, Am. Foundrymen's Soc., 61st Annual Mtg., Cincinnati, May 6-10, 1957. Trans. AFS, **65,** 1-16 (1957).

Reviews developments in gray iron, white and chilled iron, malleable iron, pearlitic malleable iron and nodular iron castings. Examples of gray iron castings include 0.4 nickel composition for automotive camshafts, 1.00 nickel iron for crankshafts, 0.2 nickel iron for cylinders and heads and austenitic types with 13.50-17.50 nickel for heat and corrosion resistant applications. Nickel-chromium martensitic white irons are used for abrasion-resistant castings such as mill liners. Applications of nodular irons shown include: 50-ton forging hammer anvil, coke-oven door, center frame for diesel engine, farm machinery castings, cross-rail casting, rolling mill rolls, diesel engine liners, rings in jet engine bearing support, and general purpose valve. Tables summarize ASTM and military specifications for nodular iron as well as world production figures. Changes in cupola and its appurtenances are reviewed. Revisions in basis water-cooled, hot-blast cupola are illustrated. Melting of nodular iron in acid cupola and work with carbide injection are considered. Comments are made on automation in foundry with particular reference to shell molding process.—INCO. 16165

8.8.5

Steps to Take in Eliminating Hot Tears. H. Chappie, National Supply Co. Foundry, **86,** No. 7, 82-84 (1958) July.

Relates experience with casting which involves almost every corrective action known to eliminate hot tears. Casting is press platen for use in aircraft plants and weighs 27,000 lbs. Following practices reduce or eliminate cracks: using sand which absorbs expansion (wood flour); multiple gating of correct thickness; favorable design, e.g., staggered ribs; use of external 1 x 6 in. chills; and most favorable carbon content (0.40).—INCO. 16786

8.8.5

Influence of Vacuum Melting on Properties of Various Materials. W. F. Moore and A. J. Kiesler. Gen. Elec. Co. Paper before European Investment Casters' Conf., Stratford-on-Avon, May 19-23, 1957. Foundry Trade J., **104,** No. 2175, 741-745 (1958) June 19.

Discussion of what occurs in vacuum furnace and what effect vacuum melting has on mechanical properties of high temperature alloys (nickel-base M252, cobalt-base J-1570, Hastelloy R-235, Waspalloy and Udimet 500), stainless steels (Types 430 and 446), steels (4340 and 52100) and pure metals (nickel and copper). Tables, graphs.—INCO. 16560

8.8.5

Vacuum Melting of Steels Improves Engineering Properties. H. C. Rezeau, Jr. SAE J., **65,** No. 10, 67-71 (1957) Sept.

Discusses improvement of engineering properties and formability through vacuum melting of bearing steels, valve and stainless steels and low-alloy steels. Graphs show longer life of vacuum-melted bearings over air-melted bearings; increase in stress-rupture life and in corrosion resistance of valve alloys 77 nickel-16 chromium-2 titanium, 25 nickel-25 chromium, and 10 nickel-10 chromium; fatigue resistance of 25 nickel-15 chromium-2 titanium melted in vacuum and in argon; and tensile ductility and tensile strength of 4340. Improvement in fatigue resistance of vacuum-melted 25 nickel-15 chromium-2 titanium alloy is probably due to absence of titanium stringers rather than exclusion of air. To illustrate improvement of formability, experimental forgings of air- and vacuum-melted 14 chromium-14 nickel-3 tungsten alloy were tested; vacuum melting eliminated cracking and folding in critical area inside neck surface of hollow-head aircraft valve. Tables summarize tensile properties and room temperature impact properties of 4340, air- and vacuum-melted.—INCO. 16307

8.8.5, 6.2.2

Solidification and Risering of Gray Iron Castnigs. C. M. Adams, Jr., M. C. Flemings and H. F. Taylor. Massachusetts Inst. of Technology. Trans. Am. Foundrymen's Soc., **66,** 369-379 (1958); Modern Castings, **34,** No. 2, 31-41 (1958) August.

Detailed conclusions of 8-year research program on solidification and risering of gray iron castings under Inco's sponsorship. Experimental data and theoretical analyses are used to delineate fundamental variables for determining shrinkage in gray iron. Natural reduction in volume accompanying cooling of gray iron from liquid to solid depends on metal chemistry. However, size and location of voids resulting are dependent not only on chemistry but also on factors which influence movement of casting surface. These are: size and shape of casting; action of atmospheric or ferrostatic pressure; characteristics of molding material; size, shape, location and thermal treatment of gates and risers. Heterogeneous expansion resulting from graphite precipitation accentuates, rather than minimizes, solidification shrinkage. Basic principles on which gray iron risering is based are discussed. Series of castings shown illustrate these principles. Graphs.—INCO. 16759

8.9 Group 9

8.9.1

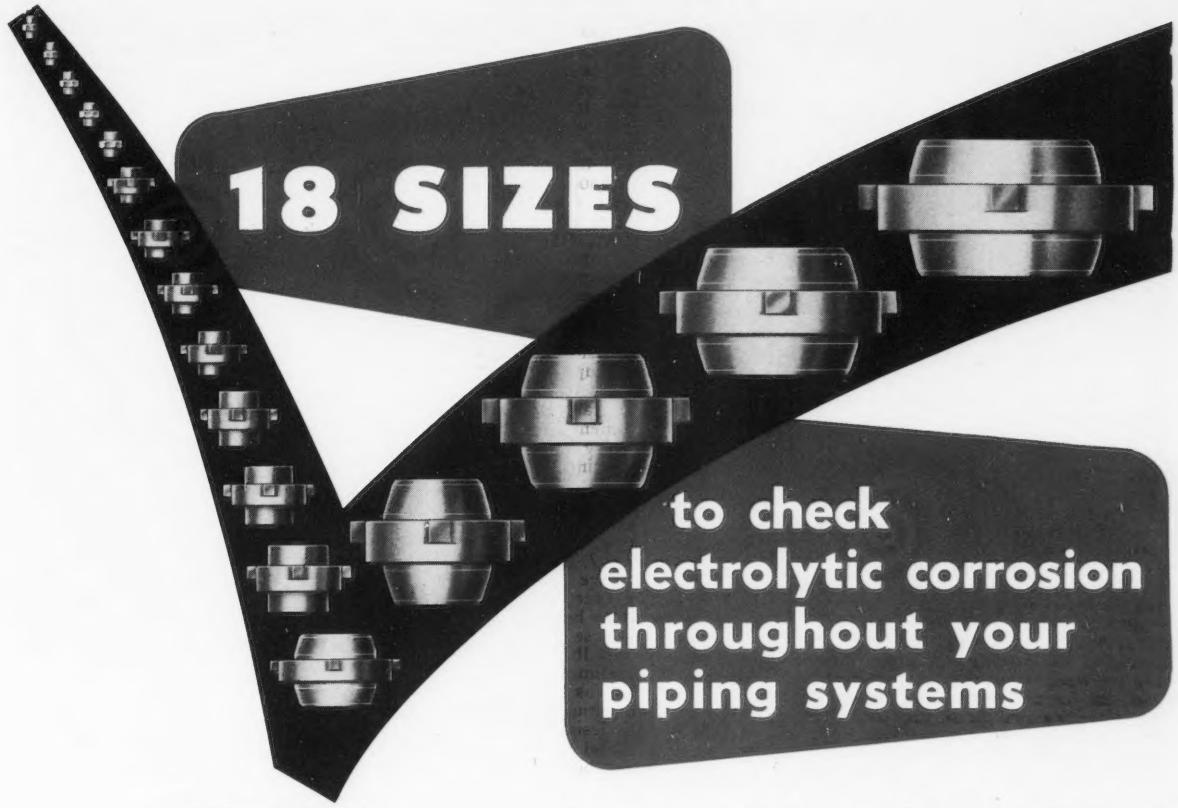
The Hot Airplane. H. B. Sipple and G. G. Wald. Paper before Am. Soc. Mechanical Engrs. Semi-Annual Mtg., San Francisco, June 9-13, 1957. Mech. Eng., **79,** No. 10, 925-927 (1957) October.

Discusses problem of suitable enduring materials for supersonic aircraft. Use of currently available heat-resisting materials imposes serious penalty on structural weight and gross weight; high-strength titanium alloys and corrosion-resisting steels now in development show promise of ameliorating weight situation. Importance of developing new fabrication and processing techniques is emphasized. Investigations should be undertaken immediately in improvement of nickel-base alloys in intermediate temperature range (under 1200 F) by variation in mill-processing and heat-treating practices. Structural weight and gross weight data, and strength/weight ratio are included for Inconel X and 17-7PH in graphic form. Chart shows general temperature limitations of common aircraft materials.—INCO. 16279

8.9.1, 3.5.3

Problems in Finishing High Speed Aircraft. R. J. E. Hunter. Paper before Chem. Inst. Canada, Protective Coatings Div., Toronto, Feb. 21, 1957. Chemistry in Canada, **9,** No. 10, 44-46 (1957) Oct.; Can. Paint and Var., **31,** No. 4, 24, 46-47 (1957).

Discussion of treatments used for protection of aluminum, magnesium and carbon steel used in construction of airplanes includes anodizing, chemical treatments, lacquering, enameling and plating. Corrosion resistant stainless steels and titanium do not require further treatment. Effect of rain erosion particularly on high speed air-craft is



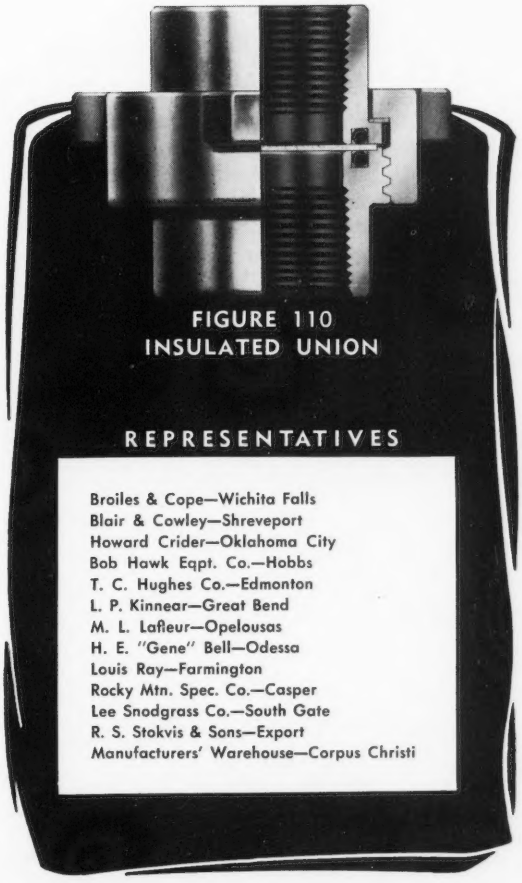


FIGURE 110
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147a

ARC-WELDING ATTACHES RISERS after acetylene torch has cut necessary tie-in holes.

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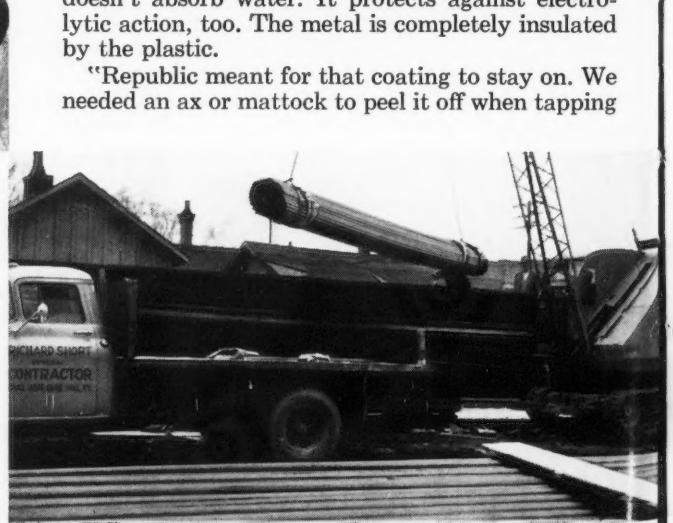
THREE-INCH X-TRU-COAT CONNECTS OLIVE HILL system to main pipeline two miles away. Two-inch lines take off from this substation and run up tree lawns of the various streets.

Contractor Richard Short tells how plastic-coated pipe solved Olive Hill, Kentucky's soil problem

"Kentucky soil is death on ordinary pipe," says Mr. Richard Short, contractor who handled installation of a new, 60,000-foot natural gas system* at Olive Hill, Kentucky. "In our part of the state particularly, soil is extremely corrosive.

"It's my business to know the materials we use. We selected X-TRU-COAT because it's downright immune to the acids and chemicals that are our problem. The polyethylene coating on this pipe doesn't absorb water. It protects against electrolytic action, too. The metal is completely insulated by the plastic.

"Republic meant for that coating to stay on. We needed an ax or mattock to peel it off when tapping



READY FOR INSTALLATION, part of the 60,000 feet of one- two- and three-inch X-TRU-COAT Plastic-Coated Steel Pipe and Tubing for Olive Hill is unloaded at railroad siding by crane.

FROM TREE LAWNS TO HOUSES, installation called for one-inch X-TRU-COAT with riser and right angle weld. Shut-off valves were attached before line entered residences.

thousands of dollars!"

in any place but at the ends of a length. Yet the same coating is really flexible. We never experienced damaged coating as a result of bending.

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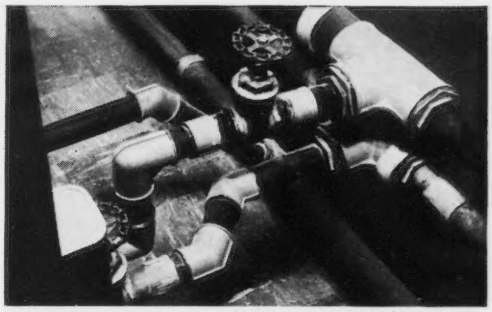

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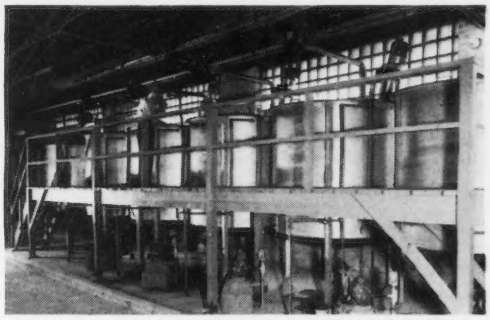

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very severe. Glass cloth reinforced plastics are especially prone to this form of erosion. One air-craft is reported to have had a plastic surface completely delaminated after two minutes flying through heavy rain, at transonic speed. A neoprene based coating was found to give satisfactory rain erosion protection to plastic glass cloth laminates at speeds to 750 m.p.h. At speeds of 1000 m.p.h. this coating breaks down very rapidly. Erosion problem will probably affect choice of metallic materials at high supersonic speeds as supersonic missiles flying for a few second through a rain storm had their aluminum alloy leading edges badly pitted. Corrosive effects of gases and deposits from machine guns, rockets, missiles and Jato are considered. Photos—INCO. 16203

8.9.5

Today's Trend in Ship Research. D. K. Felbeck. National Research Council. Welding J., **37,** No. 6, 265s-268s (1958) June.

Discusses some of significant results from current program of Ship Structure Committee, devoted exclusively to ship-hull research. Program now includes low-temperature research on fracture initiation; studies of influence of prior straining on fracture load; examination of structure of controlled-rolled steel plate; high-speed measurements of variations of strain pattern during crack propagation; and new fundamental program aimed at achieving more rational design of ships through improved understanding of interrelations between waves, ship motions, and resultant loads on and stresses in a ship. Photograph is shown of assembly of connected microcracks that form an incomplete fracture in steel specimen at —196 C.—INCO. 16519

8.10 Group 10

8.10.2

Flash Smelting Copper Concentrates. P. Bryk, J. Ryselin, J. Honkasalo and R. Malmstrom. Paper before Am. Inst. Mechanical Engrs., Annual Mtg., New York, February, 1958. J. Metals, **10,** No. 6, 395-400 (1958) June; Mining Eng., **10,** No. 6, 683-684 (1958) June.

Review of various flash smelting techniques covers reverberatory and cubic furnaces, roasting type furnace, reaction shaft and cyclone chamber. First flash smelting operation on commercial scale is Outokumpu process at Harjavalta, Finland. Oxidation reactions are carried out in vertical shaft, in downwardly flowing concurrent system. Preheated air and dried concentrate are fed into burner at top of shaft. Ignition takes place instantly. By exothermic reactions temperature of particles is raised to smelting temperature. Partial pressure of oxygen decreases to zero on leaving shaft, providing for good utilization of oxygen. Separation of particles from gas takes place when gases are turned through 90° angle. Gases containing mostly sulfur dioxide and nitrogen are exhausted from furnace at temperature slightly above slag temperature. Particles are collected in molten bath in settler. Iron oxide and silica form slag and molten matte drops are separated and collected on furnace hearth. Concentrate handling, flash smelting furnace, waste heat boiler, heat exchanger, cottrell and gas fume treatment, matte and slag treatment are described. Table gives typical analysis for concentrate, flux, matte and slag for the Harjavalta Smelter. Tests with 800 kw electric furnace show that copper content of flash smelting slag can be reduced to 0.2, while cobalt and nickel contents reach level under 0.1, which means unusually high recovery for nickel and cobalt. Mention is made of Inco's flash smelting operation at Copper Cliff, and of Furukawa Mining Co.'s copper smelter in Ashio, Japan. Diagrams.—INCO. 16454

8.10 2

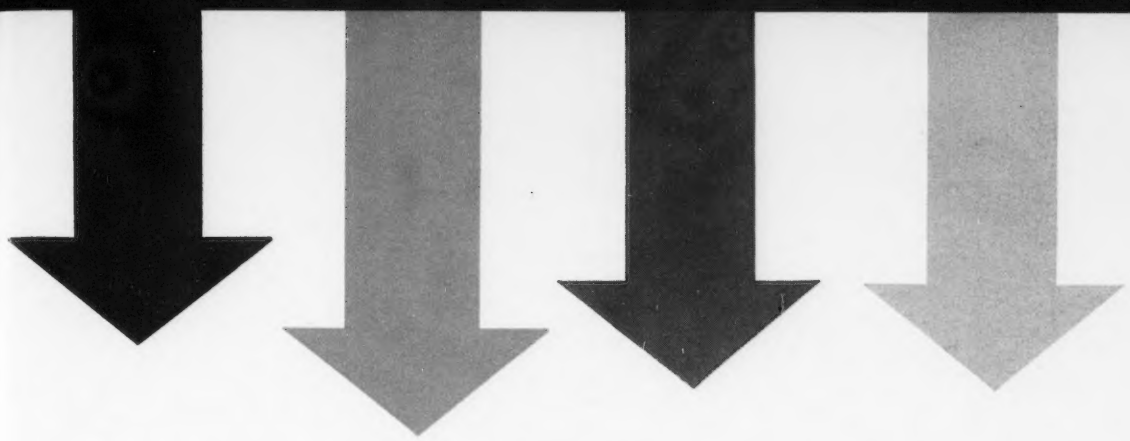
Fiber Metallurgy. R. H. Read, W. Pollack and E. W. McGee. Armour Research Foundation. Precision Metal Molding, **16,** No. 4, 25-26, 53, 58 (1958) April.

Processes for manufacturing a new class of porous metal products having a high strength as well as high permeability and a new series of composite materials such as plastics, ceramics or soft metals reinforced by fiber metal networks were developed at Armour Research Foundation. Porous metal products can be manufactured in the form of continuous sheet or in intricate shapes. Fiber metal base composites combine features of a metal network with density, refractory, corrosion or other desired properties of a second material and permit materials design for unusual service conditions. A stainless steel fiber plate and a nonporous stainless plate of about the same weight per area were in turn exposed to identical applications of an oxygen acetylene flame. The nonporous plate was burned through in about 20 seconds whereas the fiber material exhibited only surface damage after a 3 minute exposure. This preliminary test suggests many thermal barrier applications. Diagrams, graphs. —INCO. 16100

8.10.3, 8.10.2, 6.3.6

Mining in Finland. Part 4. Mining, Milling, Smelting Outokumpu Copper Ore. C. Mamen. Can. Mining J., **79,** No. 5, 73-81 (1958) May.

Description of operations of Outokumpu Oy, a state-controlled mineral empire owning deposits of copper, copper-lead-zinc and copper-nickel ore and operating a number of mines and concentrators throughout central and southern Finland. Finished products in addition to copper and copper alloy products include nickel sulfate, platinum metals and selenium. Review of methods used at Outokumpu Mine, 220 miles northeast of Helsinki, covers mining, roof bolting, haulage, crushing, concentrating and flotation (flowsheet given). Smelting of copper concentrate is done at Harjavalta using a new flash smelting process wherein additional heat required to carry on smelting process is obtained through preheating of air by using heat content of waste gases; this differs from process used by Inco at Copper Cliff since 1952 where pure oxygen supplies additional heat. Outukumpu company is presently preparing to use their second flash smelting furnace for treatment of nickel concentrates from a new nickel mine coming into early production. Illustrations, tables.—INCO. 16579



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